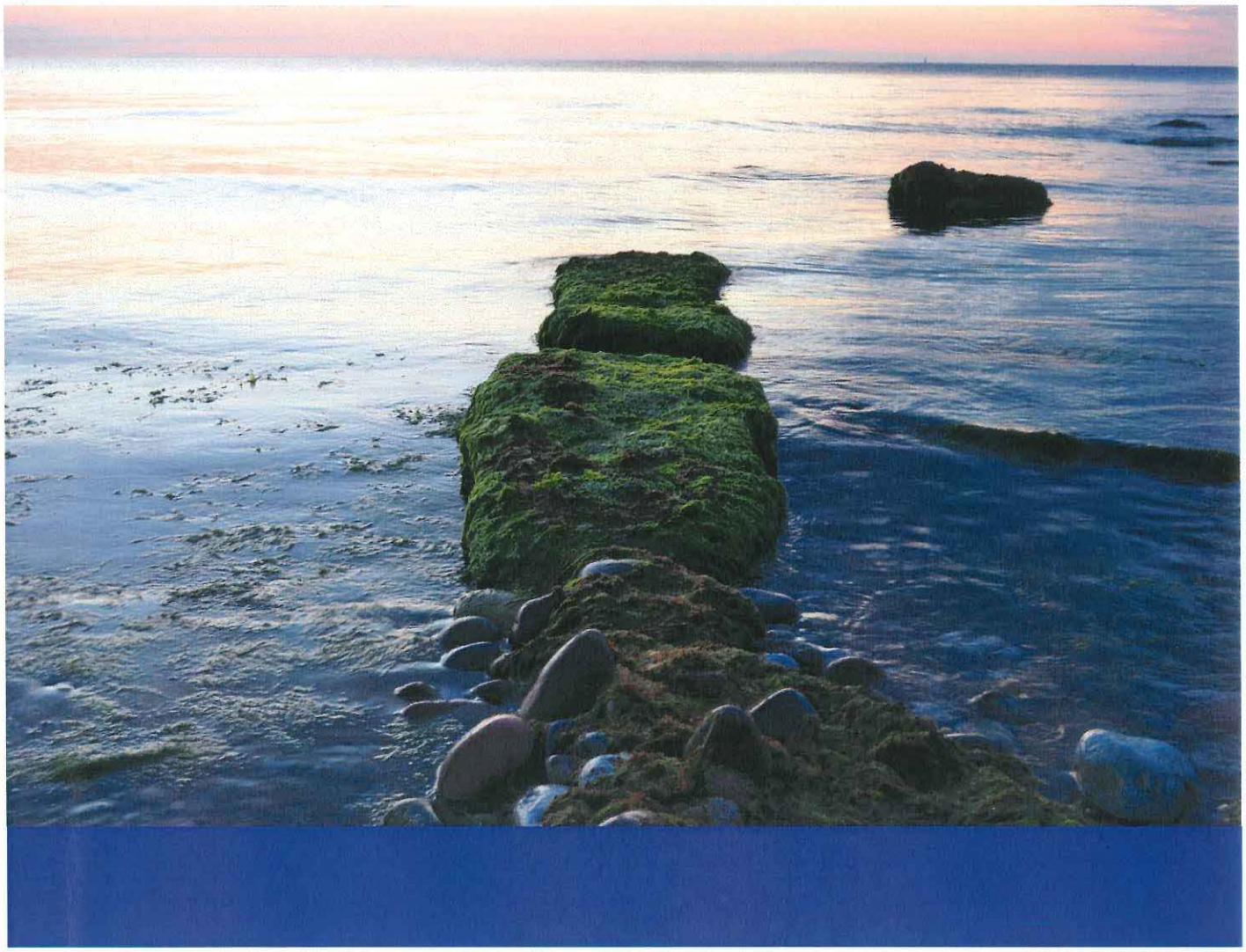


WORKPLAN TO COMPLETE SITE CHARACTERIZATION AND ADDRESS OUTSTANDING DERA ISSUES BINDER 1 OF 3

BINDER 1: WORKPLAN AND APPENDIX A

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

Via Email
April 30, 2014

Gerry Manley, CHMM
Vice President, EHS Compliance
RSR Corporation
2777 N. Stemmons Fwy., Suite 1800
Dallas, TX 75207

RE: March 28, 2014 Workplan to Complete Site Characterization and Address Outstanding DERA Issues
Quemetco, Inc.
900 Quemetco Drive
Indianapolis, Indiana
IND 000 199 653

Dear Mr. Manley:

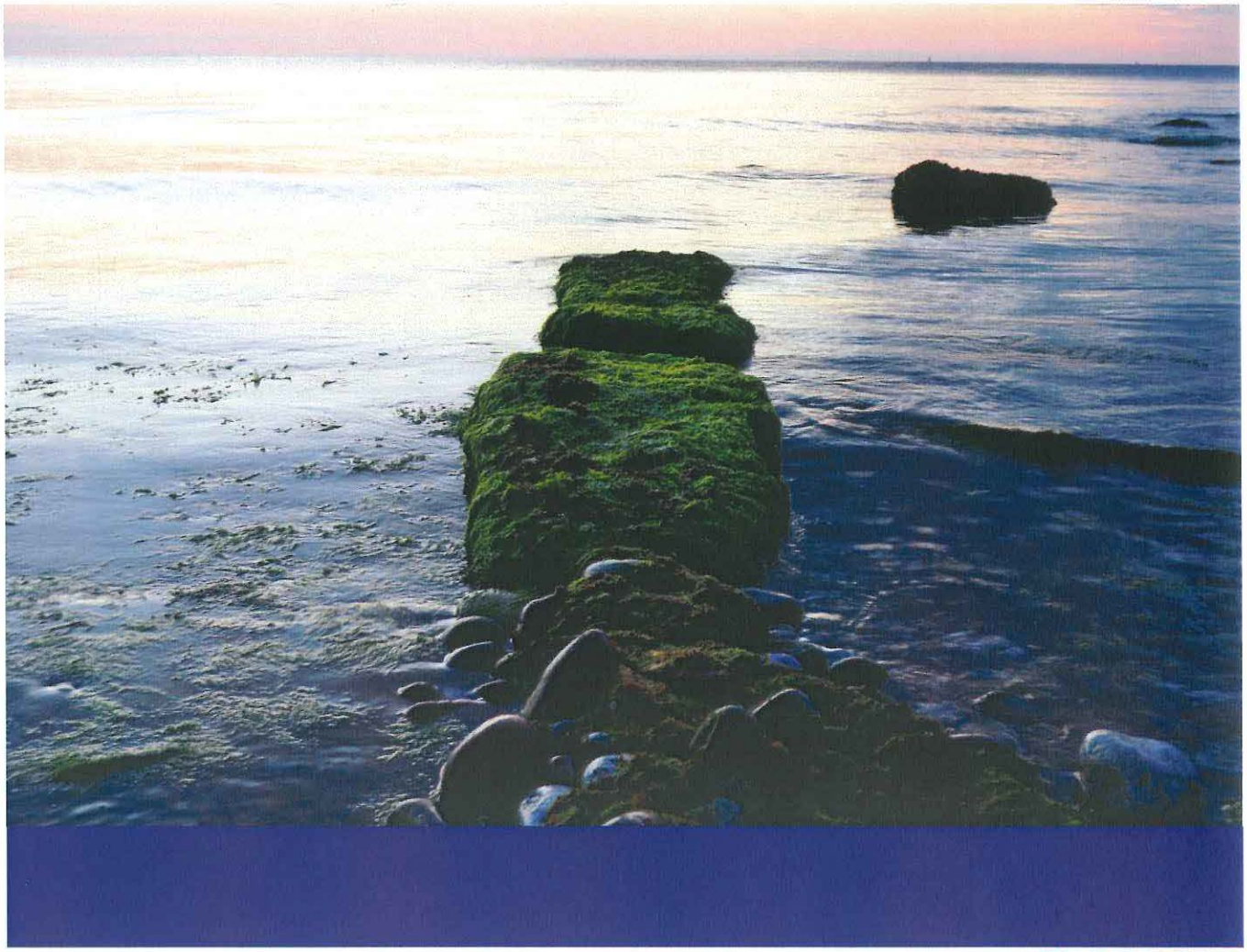
The U.S. Environmental Protection Agency has completed its review of the March 28, 2014 Workplan to Complete Site Characterization and Address Outstanding DERA Issues (Workplan) at the Quemetco, Inc. Site located in Indianapolis, Indiana. The Workplan describes the continued environmental investigations to be undertaken to determine the full nature and extent of lead contamination across the Site. It also includes the field sampling plan and analytical methods that will be used as part of the continued ecological risk evaluations occurring at the Site. EPA approves and requests implementation of the Workplan as proposed with the addition of details discussed via emails on April 9, 2014 and April 28, 2014 between EPA and Mr. Rick Freudenberger of WSP Services, Inc.

If you have any questions regarding this matter, please contact me at (312) 886-1484 or via email at dodds.jennifer@epa.gov.

Sincerely,

Jennifer Dodds
Project Manager

Cc: Rick Freudenberger, WSP



WORKPLAN TO COMPLETE SITE CHARACTERIZATION AND ADDRESS OUTSTANDING DERA ISSUES

Quemetco, Inc., 900 Quemetco Drive, Indianapolis, Indiana
US EPA Identification Number: IND 000 199 653
Revision 2, March 28, 2014

Dodds, Jennifer

From: Freudenberger, Rick E. [rick.freudenberger@wspgroup.com]
Sent: Monday, April 28, 2014 5:16 PM
To: Dodds, Jennifer
Cc: Mitton, Betsy; Waldman, Allen J.; Gerard Manley (GManley@rsrcorp.com)
Subject: RE: Quemetco - Use of XRF Data

Jennifer,

Please forgive the delay in this response. I have been out for nearly two weeks on a family medical emergency.

With respect to our telephone discussion on April 14, when we are in the field for implement the Workplan to Complete Site Characterization and Address Outstanding DERA Issues, we intend to step out from any XRF samples that exceed the Ecological Action Level in order to roughly delineate the extent of any such exceedance. This step out approach will be most rigorous within the sensitive ecological areas (along Julia Creek and near the ponds) and will also be applied to other sampling areas to the north and east of the fenced plant area.

We have put the laboratories on alert and expect to provide you with a start date for the sampling this week.

We look forward to the issuance of your approval letter. Please don't hesitate to contact me or Betsy Mitton if you have any questions or need additional information.

Rick Freudenberger

From: Dodds, Jennifer [mailto:dodds.jennifer@epa.gov]
Sent: Monday, April 21, 2014 1:22 PM
To: Freudenberger, Rick E.
Subject: Quemetco - Use of XRF Data

Rick –

I wanted to get back to you with a response on the use of the XRF data in future risk assessments. As we discussed on the phone, the XRF data is useful to help characterize the site; to determine where we have high lead levels and where we do not. However, the XRF data cannot be used in risk assessments as a part of the statistical calculations within the risk assessments.

If you need further clarification on this, please let me know and I can have one of our QA/QC experts provide a more detailed explanation.

Thank You,

Jennifer Dodds
U.S. Environmental Protection Agency, Region 5
Land and Chemicals Division
77 West Jackson Blvd, LU-9J
Chicago, IL 60604-3590
Tel: (312) 886-1484 Fax: (312) 692-2068
dodds.jennifer@epa.gov

Dodds, Jennifer

From: Freudenberger, Rick E. [rick.freudenberger@wspgroup.com]
Sent: Thursday, April 10, 2014 1:00 PM
To: Dodds, Jennifer
Cc: Mitton, Betsy; Ruth Hull; Waldman, Allen J.; Wong, Wanda; Gerard Manley (GManley@rsrcorp.com)
Subject: FW: Revised DERA Workplan, Quemetco, Inc., Indianapolis, IN
Attachments: 00007998-017 Figure 7.pdf

Jennifer,

Thanks for your timely reveiew. See responses to items 2. through 4. below.

I have forwarded comment 1. to Ruth Hull and after we connect, Ruth and I will set up a call to discuss further with you.

Rick

-----Original Message-----
From: Dodds, Jennifer [dodds.jennifer@epa.gov]
Received: Wednesday, 09 Apr 2014, 1:58PM
To: Freudenberger, Rick E. [rick.freudenberger@wspgroup.com]
Subject: RE: Revised DERA Workplan, Quemetco, Inc., Indianapolis, IN

Rick –

I've almost completed my review of your latest submittals and I do have a few questions that I'll try and detail here, however they may be more easily answered on a quick conference call.

1. The Ecological Action Level: I initially shared this concern with Ruth Hull when she first shared the Eco Action Level memo with me. It's not clear what exactly you are planning to use the Eco Action Level for? I requested its development to aid in determining where we may have ecological concerns in soils across the site, similar to how the human health action level is being used. Meaning we are continuing to characterize high lead levels in soil through XRF; stepping out if those soils exceed the 400 ppm human health action level. I was intending to use the eco action level in a similar manner as many areas of the site will not have human health concerns but rather eco concerns. If you do not intend to use the eco action level to aid in this eco characterization, how will you determine where we should stop taking XRF samples for eco concerns? Again, my intent is to avoid having to re-sample to develop a CMS. If we've stopped characterizing an area because it reached a human health action level of 400 ppm, but we later determine that the area needs to be cleaned up to an ecological action level of 200ppm, for example, we wouldn't know where the clean up should end. We would need to re-sample. Hence the development of the eco action level. It's not necessarily cleanup level; it's a stopping point for characterizing the site based on eco concerns. Please explain.

2. Sediment Field Parameters: Table 1 of the Workplan lists a more comprehensive set of the sediment field measurements/parameters than are detailed in your March 27, 2014 response memo. Is Table 1 correct or the March 27, 2014 response memo? ***The memo is correct. Table 1 indicates the laboratory analyses for sediments. The field measurements/parameters to be collected for sediments is included in the memo and also described in Section 3.4.1, page 16 of 24, of the SAP (Appendix A).***

3. Figure 7: Section 3 of the DERA Workplan refers to a Figure 7 which shows all of the proposed sampling locations for the DERA. I am unable to locate Figure 7. ***It should have been included in the electronic copy. It is***

WORKPLAN TO COMPLETE SITE CHARACTERIZATION AND ADDRESS OUTSTANDING DERA ISSUES

Quemetco, Inc., 900 Quemetco Drive, Indianapolis, Indiana
US EPA Identification Number: IND 000 199 653
Revision 2, March 28, 2014

Prepared on behalf of:

RSR Corporation
277 Stemmons Freeway, Suite 1800
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Richard Freudenberger
Allen Waldman

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1 Introduction

The following Site Characterization and Outstanding Issues for the Detailed Ecological Risk Assessment (DERA) Workplan was prepared by WSP (previously known as WSP Environment & Energy, WSP Environmental Strategies and Environmental Strategies Corp.) on behalf of Quemetco, Inc. and describes proposed environmental investigation plans at Quemetco's facility (Site) located at 900 Quemetco Drive, Indianapolis, Indiana (Figures 1 and 2). This workplan incorporates comments provided by the U.S. Environmental Protection Agency Region 5 (U.S. EPA) in several meetings and correspondence since the workplan was originally submitted on May 19, 2008. The submittal of this revised workplan was delayed until an X-Ray Fluorescence (XRF) soil characterization study was completed as recommended by the U.S. EPA in their October 08, 2009 letter to Quemetco. The XRF investigation was conducted in April and October 2010, and December 2011, in accordance with the approved workplan submitted to the U.S. EPA on February 17, 2010. The final XRF investigation report was submitted to the U.S. EPA on March 23, 2012.

As requested by the U.S. EPA in their October 8, 2009 letter, this Workplan is intended to satisfy two objectives. These include: 1) to satisfactorily address the U.S. EPA's specific comments regarding the Ecological Investigation and Quality Assurance procedures presented in the May 19, 2008 workplan; and 2) further delineate the nature and extent of lead contamination across the Site. The completed XRF investigation, which followed the strategy presented by U.S. EPA in their October 8, 2009 letter, delineated the lateral distribution of lead in surficial and shallow soils across a large portion of the Site surrounding the manufacturing facility including property east of the facility belonging to Carrier Corporation.

The workplan is organized as follows:

- Section 1 provides an introduction and brief history of the Quemetco facility.
- Section 2 presents the Site sampling plan to further delineate lead and other constituents of concern across the Site and supplements previous phases of work to fill in data gaps. The work will include additional XRF soil testing and verification sampling, and discrete soil sampling to delineate vertical limits of lead in areas of the Site where the lateral distribution has already been defined. In addition, U.S. EPA has requested one round of groundwater sampling from the existing groundwater monitoring well network to assess the current conditions of groundwater.
- Section 3 presents the proposed DERA field sampling plan and analytical methods. The proposed sampling will include toxicity tests on soil, sediments, and surface water. Additionally, the plan for revising the DERA is presented.
- Appendices include the Sampling and Analysis Plan (SAP), Quality Assurance Project Plan (QAPP), and the Health and Safety Plan (HASP) for the entire program.

1.1 Location

Quemetco, Inc., a subsidiary of RSR Corporation, operates a secondary lead smelting facility located at 900 Quemetco Drive, in Indianapolis, Marion County, Indiana (Figure 1). The operating facility occupies eight acres.

The Site is located in an area of mixed industrial, residential, and agriculture land use. The immediate area consists of undeveloped woodlands and land zoned for industrial use. Property zoned for residential use is located approximately 0.5 mile northeast, northwest, and southwest of the facility. North of the facility are the railway line and plant for the Power Transmission Company and to the northeast is the West Park View residential area. East of the Site are farmland, a furnace and air conditioning manufacturing facility, and a freight carrier company. The area west of the Site includes the Pennsylvania Railroad and farmland. To the south is Heritage Environmental, a hazardous waste treatment facility.

1.2 Facility History

The Quemetco facility was purchased from St. Joe Minerals in 1972 as a newly constructed facility. The Site was used as farmland before the plant's construction. Quemetco recycles lead primarily from lead-acid batteries, although other lead-bearing materials are occasionally processed. Materials to be smelted (recycled) arrive by truck or rail car and are unloaded within the fenced area of the plant. The basic components of the secondary lead smelting process include breaking and crushing of batteries followed by separation of the lead, and other materials (rubber, polypropylene) in sink/float cells. The lead is separated from the rubber/plastic casings before continuing on through the smelting process in the main plant building. The lead is then conveyed to a reverberatory furnace for lead recovery to generate lead bullion. The polypropylene is washed, rinsed, and sold to various plastic fabricating facilities. The slag generated from the reverberatory furnace is processed further in the electric arc furnace to recover additional lead bullion. Lead bullion is processed in the refinery into various lead alloys according to specific customer requirements. These products are marketed as 25- to 65-pound ingots and 1-ton blocks.

Nonhazardous wastes generated at the facility include general trash, slag from the electric arc furnace, small amounts of waste oil and spent solvents and plastic battery casings and scrap metal. The waste oil and solvents are sent offsite for recycling. The plastic and scrap metal generated at the facility are also sent to offsite recycling facilities.

Other wastes generated from the lead smelting process include baghouse dusts, wastewater treatment sludge, and lead dross. The majority of this material has valuable lead content and is processed in the reverberatory furnace. Polypropylene is sold for recycling. Additional hazardous wastes generated at the facility include petroleum naphtha generated from maintenance activities and refractory brick.

In 2011, Quemetco began construction of a wet electrostatic precipitator (WESP) unit that adjoins the plant on the northeast side of the Facility (Figure 2). The WESP was completed and brought on line in March 2012. The WESP site occupies approximately 0.8 acres of land which was previously densely vegetated. Before construction of the WESP, the vegetation on the construction site was cleared and Quemetco removed soil that contained total lead concentrations above the industrial action level of 970 parts per million (ppm). The workplan for the confirmation soil sampling beneath the construction site was submitted to the U.S. EPA on October 13, 2010.

2 Site Characterization and Groundwater Sampling Plan

This section presents the sampling plan designed to further characterize the lateral and vertical extent of lead in soil across the Site and establish current groundwater conditions and a summary of the Site characterization and groundwater sampling activities is included on Table 1. The approach to soil characterization is to supplement data from previous sampling events, including the recent XRF soil investigation, and fill in data gaps where the distribution of lead in the environment is unknown. The objective will be to delineate soil lead concentrations laterally and vertically to below the U. S. EPA Region 9 regulatory screening level (RSL) for lead in residential soil of 400 milligrams per kilograms (mg/kg) (U. S. EPA, May 2012). The rationale for using the RSL is that the XRF results are being compared to a promulgated screening concentration protective of children playing on bare soil. The XRF results are not being compared to the 2003 Region 5 ecological screening levels (ESLs) of 0.05 mg/kg due to the fact that this value is below the background concentrations of lead in soil and approximates the laboratory reporting limit for lead. At the request and with the approval of U.S. EPA, an ecological-based Action Level has been developed for lead. In a memo from Intrinsik Environmental Sciences to U.S. EPA, dated February 14, 2014, it was determined that the overall Ecological Action Level for lead is 130 mg/kg (Intrinsik Environmental, 2014). It was agreed that the Ecological Action Level will not be used to screen the data and that any proposed remediation criteria will likely be based on the ecological risk assessment and not this Ecological Action Level.

Figures 3A, 3B, and 3C present a compilation of lead concentrations in soil samples collected between 1987 and 2011 including the most recent XRF data collected in 2010 and 2011. On these figures, soil concentrations above 400 mg/kg are highlighted in yellow. The figures indicate that further lateral delineation is still required to the north and northeast of the plant area. Further delineation to the east of the plant is not possible due to structures on the Carrier Corporation property. Historical data on Figures 3A, 3B, and 3C from previous exploratory borings indicate that in many areas of the Site, the vertical extent of lead has been delineated and that lead concentrations typically attenuate within five feet of the ground surface. Particular borings where vertical delineation has been achieved are identified in Figures 6A, 6B, and 6C. Five boring locations are being proposed to confirm vertical extent of lead; three on the eastern side of the plant area and two on the northern side of the plant area (Figure 6A).

The current groundwater conditions will be assessed by sampling the existing groundwater monitoring wells located as described in Section 2.2 on page 8.

2.1 Soil Sampling Plan

2.1.1 XRF Sampling Locations and Procedures

To complete the lateral delineation of lead concentrations, WSP will use the same XRF sampling strategy and methodologies that were implemented in the 2010/2011 soil investigations. This approach follows the U.S. EPA's approach presented in their letter dated October 8, 2009 and was described in WSP's *XRF Sampling and Analyses Plan* dated February 19, 2010. The strategy involves creating a grid composed of 70 foot by 70 foot squares within the target areas of the Site. Soil samples are collected for XRF analysis (0 to 1 inch below ground surface (bgs)) from within each grid square. XRF readings are measured in deeper soils (1 to 6 inches bgs) in alternate grid squares, resulting in half of the gridded area being characterized to a depth of 6 inches bgs. Also, in compliance with the U.S. EPA's recommendation, five percent of the total XRF screening samples will have fixed-laboratory verification samples analyzed for lead using SW-846 guidance method (e.g.6020). The fixed-laboratory

verification samples will only be collected from the deeper soils (1-6 inches bgs) encountered in alternate grid squares during XRF screening. Verification soil samples will be analyzed by Pace Analytical Services, Inc. (Pace Analytical) of Indianapolis, Indiana. Pace Analytical is accredited by the National Environmental Laboratory Accreditation Program (NELAP). The details of the investigative procedures are presented in the SAP (Appendix A). Information regarding Pace Analytical is included in the QAPP (Appendix B).

The proposed XRF sampling locations are represented by the empty grid squares north and northeast of the plant area shown on Figure 5. XRF readings/samples will be taken first in the row of grid squares nearest the plant labeled 267 through 288. If an XRF reading in a grid square exceeds 400 mg/kg lead, then the investigation will extend into the select grid square immediately adjacent to the grid square where the XRF reading was above 400 mg/kg. Thus, additional XRF readings may only be collected in specific squares and not necessarily an entire row, as applicable. XRF sampling will continue laterally until the lead concentrations have been delineated to below 400 mg/kg.

XRF sampling will extend to the Quemetco property line to the north and northeast. Quemetco has obtained an access agreement from Carrier Corporation; therefore, if necessary, the investigation may extend onto the Carrier Corporation property. The sample grids on the Carrier Corporation property will be extended as far as possible but not beneath any buildings or structures. If XRF sampling extends to the Carrier Corporation property, the pattern of collecting deeper samples at 6 inches from alternate grid squares and collecting verifications samples for laboratory analysis will continue. Each sample location will be recorded using a global positioning system (GPS) device. A map depicting all sample locations and surrounding areas will be provided with the final report. WSP will notify U.S. EPA at least 48 hours before any scheduled fieldwork.

2.1.2 Direct Push Soil Sampling Procedures

To satisfy U.S. EPA's requirement to delineate the vertical extent of lead contamination in soil, WSP will advance five soil borings and collect samples to a depth of 8 feet bgs in areas of the Site where additional data is needed. The proposed boring locations (VD-1 through VD-5) are shown on Figure 6A.

A direct push drilling rig (GeoProbe® or equivalent) will be used to advance 3.5-inch outside diameter dual-tube drill rods equipped with Lexan or acetate (or equivalent) sleeves. Dual tube sampling equipment is preferred because the outer rods remain in the ground to prevent soil sloughing when the sampler (inner tube) is removed. To collect a continuous soil core, the sampler will be pushed in 4-foot intervals. Soil samples for laboratory analysis will be collected from each boring at depth intervals of 1 to 1.5 feet bgs, 3 to 3.5 feet bgs and 7 to 7.5 feet bgs.

Sampling personnel will wear disposable nitrile gloves and change gloves before collecting each sample to prevent cross-contamination. The desired sleeve(s) will be covered with Teflon tape, capped and labeled. Sample handling procedures are described in the SAP presented in Appendix A.

Upon completing the soil collection procedure, the borehole will be backfilled with hydrated bentonite chips. Borings drilled in paved areas will be finished to grade with a cement cap. Detailed procedures for soil sampling using a Geoprobe rig are provided in the SAP (Appendix A).

Samples will be analyzed for lead using SW-846 guidance method (e.g., 6020). The lab will be instructed to analyze the shallowest samples from each boring first and only analyze the next deepest sample if the first sample exceeds the 400 mg/kg lead threshold. Specific analytical and laboratory procedures are presented in the QAPP (Appendix B).

Each boring location will be surveyed using a portable GPS unit. A map depicting all sample locations and surrounding areas will be provided with the final report. WSP will notify U.S. EPA at least 48 hours before any scheduled fieldwork.

2.2 Groundwater Monitoring Well Sampling Plan

A total of 13 monitoring wells (MW-1 through MW-13) and nine piezometers (PZ-1 through PZ-9) were installed throughout the property to characterize the groundwater quality at the Site (Figure 2). A well reconnaissance conducted in December 2011 confirmed that all the existing wells are 2 inches in diameter and are approximately 24 to 48 feet in depth. Wells MW-11 and MW-12 could not be located and are presumed destroyed. Well MW-1 was replaced with MW-1R. Well MW-2 has a submersible pump stuck inside of it and could not be measured. Two of the wells, MW-1R and MW-6, have silted in by 9 to 10 feet of material, respectively. MW-3 has silted in by approximately 3 feet of material. Monitoring wells MW-4, 5, 7, 8, 9, 10, and 13 were most recently sampled in 1999 and did not appear to have silt in them in 2011. Historical total depth measurements or well construction details for MW-13 and the nine piezometers were not available to compare the recent total depth measurements.

From August 1996 to August 1997, four quarterly groundwater sampling were conducted by Environmental Strategies Corp (now known as WSP) as part of the Phase II RFI at the Site. The samples were analyzed for total metals, dissolved metals, phenols and total suspended solids. Additionally, one round of groundwater sampling was collected from the monitoring wells and PZ-1 in May 1999. Analytical results from 1996 and 1997 were compared to regulatory screening criteria (human health and ecological screening levels) or background levels for the constituents of concern and indicated lead, arsenic, antimony, cadmium, and chromium are the only constituents that had concentrations exceeding the screening levels at the time (Final Remedial Investigation Report and Risk Assessment, Quemetco, Inc., Indianapolis, Indiana, prepared by Environmental Strategies Corporation, July 30, 1999).

Groundwater samples collected from MW-8 in 1996 to 1997 typically contained the highest metal concentrations in groundwater at the Site. Analytical results from 1999 indicated that one well, MW-12 (which is no longer present), exceeded the human health and ecological screening levels. None of the constituents in MW-8 exceeded screening levels.

At the request of U.S. EPA, WSP proposes to collect one round of groundwater samples from monitoring wells MW-1R, MW-2 through MW-10 and MW-13 to obtain updated information on groundwater quality beneath the Site. None of the piezometers will be sampled.

2.2.1 Well Development

Since it has been over 10 years since the wells were sampled and because some of the wells have more than 3 feet of silt on the bottom, each well will be developed prior to sampling. Although the piezometers will not be sampled, they will be developed in order to use them for water level measurements. The wells and piezometers (MW-1R, MW-2 through MW-10, MW-13, and PZ-1 through PZ-9) will be developed using bailing, surging, or pumping techniques until the effluent water is relatively free of fine sediment and the total depth of the wells have been restored to the depths recorded in 1999. This work will be performed by a local drilling subcontractor and supervised by WSP. The well development process is described in the SAP in Appendix A.

The drilling contractor will also attempt to retrieve the stuck pump from well MW-2. If this proves to be unsuccessful, piezometer PZ-1 will be developed and sampled in place of MW-2 since it is of similar depth and they are proximal to each other (Figure 2).

2.2.2 Groundwater Sample Collection and Analysis Procedures

The groundwater monitoring wells will be purged and sampled using low-flow minimal-drawdown sampling techniques in general accordance with ASTM standard D6771-02, *Standard Practice of Low-Flow Purging and Sampling for Wells and Devices Used for Groundwater Quality Investigations* and WSP's Standard Operating Procedures (SOPs) presented in the SAP (Appendix A). The general sample collection procedures are described below.

- A complete round of water levels will be measured from all the wells and piezometers (i.e., MW-1R, MW-2 through MW-10, MW-13, and PZ-1 through PZ-9) at the start of the sampling event. Water level measurements will be used to determine the direction of potential groundwater flow, document water level changes, calculate purge volumes, and to ascertain when water levels have recovered sufficiently after purging to perform sampling.
- For wells MW-1R, MW-2 through MW-10, and MW-13, a submersible pump will be lowered very slowly into the well and positioned approximately five feet off the well bottom to ensure the pump intake is within the middle portion of the monitoring well screen interval. The well will be allowed to stabilize for 15 minutes before starting the purge.
- Water shall be purged from each monitoring well until a representative sample of the groundwater from the water-bearing zone can be collected. Purge rates shall be as slow as practical; ideally no faster than the rate that water re-enters the casing. The starting purge rate will be as slow as possible to initiate flow but can be increased to as high as 500 milliliters per minute (mL/min) if it is established that the water level is stable (drawdown is less than 0.3 feet). Purging shall continue until groundwater indicator parameters that will be measured by the field technician have been stabilized. The parameters will include pH, electrical conductivity, temperature, oxidation-reduction potential, and dissolved oxygen.
- Samples with minimal physical change from conditions in the formation are desired. To that end, samples will be taken of groundwater that has moved into the well casing with minimal disturbance and brought to the surface with minimal contact with outside air. Samples will be collected via the discharge tubing from a submersible pump.
- Samples will be collected in clean, pre-preserved laboratory-supplied containers. Containers will be clearly labeled by site, well or identification number, preservative type and amount, date, and analysis requested. The field technician will complete a chain-of-custody form to account for each sample.
- All sampling equipment will be thoroughly decontaminated before the commencement of work and between each sampling location to prevent potential cross-contamination. Decontamination will involve washing the equipment with non-phosphate detergent and rinsing with tap water.

Detailed well sampling procedures including sample collection, sample handling, decontamination, and field documentation and are provided in Table 2 of this Workplan and also in WSP's SOPs presented in the SAP (Appendix A).

The groundwater samples will be analyzed for total and dissolved metals by EPA methods 6020/7470A series; total suspended solids by SM2540D; and total phenols by EPA method 8270C.

3 DERA Field Sampling Plan

This section presents the proposed DERA field sampling plan and analytical methods. The DERA field sampling will include benthic invertebrates and animal toxicity tests on soil, sediments, and surface water. A summary of the proposed DERA sampling and analyses are provided in Table 1. The proposed sampling locations for the DERA are shown in Figure 7.

3.1 Intended Data Usage

Toxicity testing results will be used in the Effects Assessment component of the DERA for the various receptors that have multiple lines of evidence in Julia Creek, the Ponds and surrounding areas (i.e., earthworms, fish, benthic invertebrates). The addendum to the DERA will be completed using the new data collected and will assess new receptors (i.e., Indiana bat, sandpiper) and reassess the risks to the American robin by incorporating the new data. These results will be incorporated into the weight-of-evidence evaluation in the Risk Characterization component of the DERA addendum and will also include updating exposure parameters and toxicity reference values, as needed.

Results from the chemical analyses for sediment, soil, and surface water will be used to supplement the ecological-biological-toxicology results. Chemical results for soils will not be compared to ecological screening levels for soils (EcoSSLs). The agreed upon Ecological Action Level for lead for the Quemetco site (130 mg/kg for birds, 490 mg/kg for mammals) will be used for screening and will be further assessed in the addendum to the DERA. DERA's generally use the lowest-observed-adverse-effect levels (LOAELs) for determining the need for remediation. Therefore, LOAELs have been used to derive Ecological Action Levels for the Quemetco site (Intrinsik memo to U.S. EPA, February 2014). The Ecological Action Levels for the Quemetco site were derived by using the toxicity data for wildlife compiled within EPA's Eco-SSL document (EPA, 2005). The DERA addendum will assess lead risks to designated wildlife receptors using more recent and updated exposure and toxicity assumptions. Specific quality control and quality assurance procedures are described in the QAPP, which is included as Appendix B.

3.2 Ponds and Julia Creek Soil Sampling

In 2006, soil samples were collected from around Ponds No. 1, 2 and 3 including three samples from Pond No. 1, three samples from Pond No. 2, and two soil samples from Pond No. 3. Concentrations of lead ranged from 1,900 mg/kg to 2,910 mg/kg around Pond No. 1; from 705 mg/kg to 3,190 mg/kg around Pond No. 2; and from 28.6 mg/kg to 141 mg/kg around Pond No. 3. U.S. EPA indicated that additional soil samples must be taken around each pond (per the August 10 2007 letter to the U.S. EPA, an additional five soil samples will be taken from around the perimeter of Pond No. 3). Therefore, an additional four soil samples (0 to 6 inches bgs) will be collected from around Ponds No. 1 and 2, and five soil samples will be collected from around Pond No. 3 (Figure 7), for a total of 13 additional soil samples. Samples P1-13 through P1-16 will be collected from Pond No. 1; samples P2-10 through P2-13 will be collected from Pond No. 2; and samples P3-16 through P3-20 will be collected from Pond No. 3.

As requested by the U.S. EPA, soil sampling will be conducted to the east of Pond No. 1 and Pond No. 2 where soils may have been impacted by lead in the Northeast area described in Section 2.1.1 on pages 6 and 7. Soil sample P1-16 will be collected from the east side of Pond No. 1, and P2-11 and P2-12 will be collected from the east side of Pond No. 2.

For Pond No. 3, three soil samples will be collected from the south and east side of the pond, and the swale (P3-16, P3-19, and P3-20) and the remaining two soil samples (P3-17 and P3-18) will be collected on the north side of the pond (Figure 7).

In 2006, three soil samples were collected along Julia Creek. Lead concentrations ranged from 317 mg/kg to 846 mg/kg. An additional 15 soil sampling locations are proposed along Julia Creek: three upstream locations (JCS-1, JCS-2 and JCS-3); one adjacent to the Site (JCS-4); eight along the creek on the site property JCS-5 through JCS-8 and JCS-18 through JCS 21); and three offsite downstream locations (JCS-9, JCS-22 and JCS-23). Soil sampling locations along Julia Creek are identified on Figure 7.

The soil samples will be collected from 0 and 6 inches bgs and will be analyzed for metals, pH, total organic carbon (TOC), soil moisture, water holding capacity and cation exchange capacity (CEC).

3.2.1 Soil Sample Collection and Analysis Procedures

Based on historical data from the Site, the target parameters for chemical analysis are lead, antimony, arsenic, barium, cadmium, chromium, copper, lead, mercury, nickel, and zinc. Based on current and historical operations conducted by RSR, other metals found in Appendix IX for RCRA metals analysis have been eliminated (Ecological Risk Assessment, October 19, 2006, prepared by Cantox and WSP Environmental Strategies).

Procedures for collecting the soil samples, field notebook records, and decontamination activities are presented in the SAP (Appendix A). Details on the specific analyses for each sample collected is presented in Table 1 of this Workplan and also included as Table B1 in the QAPP presented in Appendix B.

3.3 Sediment Samples

Sediment samples will be collected from Pond Nos. 1, 2, and 3 and Julia Creek. The sediment sampling locations are included on Figure 7. Two sediment samples will be collected from Pond No. 1 (SD-9 and SD-10); three sediment samples will be collected from Pond No. 2 (SD-11, SD-12 and SD-13); and four sediment samples will be collected from Pond No. 3 (SD-14 through SD-17). All of the sediment samples collected from the ponds will be analyzed for metals, total phenols, AVS/SEM, interstitial pore water-ammonia, interstitial pore water-pH, TOC, particle size analysis and CEC. One sediment sample from each pond (SD-10, SD-13, and SD-17) will be used for toxicity testing including the 28-day toxicity test using *H. azteca* and the 10-day toxicity test using *C. dilutus* (formerly *C. tentans*).

Fourteen (14) sediment sampling locations in Julia Creek are identified as SD-1 through SD-8 and SD-18 through SD-23. All 14 sediment samples will be analyzed for metals, total phenols, AVS/SEM, interstitial pore water-ammonia, interstitial pore water-pH, TOC, particle size, and CEC. Eight of the 14 samples collected in Julia Creek (SD-1, SD-3, SD-4, SD-5, SD-18, SD-20, SD-22 and SD-23) will be used for toxicity testing including the 28-day toxicity test using *H. azteca,* and the 10-day toxicity test using *C. dilutus*.

Sediment samples will be transported to Pace Analytical for chemical analysis and to Northwestern Aquatic Sciences (NAS) for 28-day toxicity testing using *H. azteca* and 10-day toxicity testing using *C. dilutus*. All samples will be shipped to the laboratories by an overnight mail service or by courier.

Procedures for collecting the sediment samples, field notebook records, and decontamination activities are presented in the SAP (Appendix A). Details on the specific analyses and quality control procedures for each sample collected are presented in Tables 1 and 2 of this Workplan and also included as Table B1 in the QAPP (Appendix B).

3.4 Surface Water Samples

Based on the results of sampling and analysis from 2006, two additional surface water samples from Pond No. 3 will be collected for chemical analysis and toxicity testing. The surface water samples from Pond No. 3 will be chemically analyzed for total and dissolved metals, pH, and water hardness by Pace Analytical. Toxicity testing on the two surface water samples from Pond No. 3 (P3-SW-1 and P3-SW-2) will include *P. promelas* (fathead minnow) and *C. dubia*. Surface water samples collected from Pond No. 3 are indicated on Figure 7.

Fourteen (14) surface water samples will be collected from Julia Creek and used for chemical analysis. Six surface water sampling locations will be placed north of the NPDES sampling point (SW-1 through SW-5 and SW-18). Six surface water sampling locations will be placed approximately every 200 feet between the NPDES outfall and SW-8 (SW-6, SW-7, SW-8, SW-19, SW-20, and SW-21). Two additional surface water samples (SW-22 and SW-23) will be collected further downstream of SW-8 (approximately 500 feet and 1,000 feet from SW-8) in order to adequately characterize the conditions of Julia Creek. The 14 surface water sampling locations are indicated on Figure 7 as SW-1 through SW-8 and SW-18 through SW-23. The Julia Creek surface water samples will be chemically analyzed for total and dissolved metals, pH, and water hardness by Pace Analytical.

As noted in the DERA report, additional samples of Julia Creek water are recommended for *P. promelas* and *C. dubia* testing and chemical analysis. A total of eight surface water samples will be collected from Julia Creek for toxicity testing using *P. promelas* and *C. dubia.* The surface water sampling locations will attempt to capture the full range of lead concentrations found in Julia Creek. The surface water sampling locations that will be used in the toxicity testing include SW-1, SW-3, SW-4, SW-5, SW-18, SW-20, SW-22 and SW-23.

Surface water samples will be transported by overnight mail service or courier to Pace Analytical. Surface water samples collected from Pond No. 3 and Julia Creek will be used for toxicity testing by NAS. The surface water samples will be transported to NAS by overnight mail service.

Procedures for collecting the surface water samples, field notebook records, and decontamination activities are presented in the SAP (Appendix A). Details on the specific analyses for each sample collected are presented in Table 1 of this Workplan and also included as Table 1-1 in the QAPP (Appendix B).

3.5 Earthworm Toxicity Testing

There is a potential for earthworms to be impacted by metals in soil in selected areas of the Site. Earthworms and other soil invertebrates serve many ecologically important functions, including aeration of the soil, breakdown of refuse, and food for mammalian and avian wildlife. To evaluate the toxicity of metals to earthworms, soil samples (0 to 6 inches bgs) will be collected around Pond Nos. 1, 2, and 3, Julia Creek, the Slag Pile, the NE Area, and a reference area near the 2006 background sampling locations on the southwest side of the Site. A composite sample will be taken from each of these seven

locations. The soil will be used in the evaluation of earthworms using U.S. EPA protocol 600/3-88-029, a 14-day toxicity test that evaluates survival and biomass of earthworms.

The locations of the soil samples collected for earthworm toxicity testing are identified as COMP-1 through COMP-3, COMP-JC, COMP-SP, COMP-NE and COMP-BK and include the following:

- Soil sample COMP-1 will be a composite sample collected from soil sampling locations P1-13 through P1-16 around Pond No. 1.
- Soil sample COMP-2 will be a composite sample collected from soil sampling locations P2-10 through P2-13 around Pond No. 2.
- Soil sample COMP-3 will be a composite sample collected from P3-16 through P3-20 around Pond No. 3.
- Soil sample COMP-JC will be a composite sample from four locations along Julia Creek including JCS-4 through JCS-7.
- Soil sample COMP-SP will be a composite sample collected from COMP-SP1, COMP-SP2, COMP-SP3 and COMP-SP4.
- Soil sample COMP-NE will be a composite sample from the NE Area and will consist of soil sample locations COMP-NE1, COMP-NE2, COMP-NE3 and COMP-NE4.
- Soil sample COMP-BK will be a composite sample from the southwest area of the Site including BSB-1, BSB-9 and BSB-10.

All composite soil sampling locations are included on Figure 7.

Earthworm toxicity testing will be conducted by NAS. Detailed soil sampling procedures are provided in the SAP (Appendix A).

3.6 Benthic Invertebrate Testing

There is a potential for effects of metals on benthic invertebrates. The spotted sandpiper and other wading birds, including the mallard duck, consume benthic invertebrates. As requested by U.S. EPA, benthic invertebrates will be collected from Julia Creek and the Ponds for tissue analysis and used in the risk characterization for the Spotted sandpiper and American robin. Benthic invertebrate samples will be collected from sample locations BI-1 through BI-5, BI-8, BI-9, BI-12, BI-13, BI-15, BI-17 through BI-20, BI-22 and BI-23 (Figure 7). The benthic invertebrate samples will be shipped to Pace Analytical and chemically analyzed for total metals in tissue and moisture content by EPA methods 6020/7471 and ASTM D 2974-87.

Table 1-Site Characterization and DERA Sample Collection and Analysis

Sample Matrix	Analysis	Number of Samples	Sample ID (a)	Method
soil	Metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn)	28	P1-13 through P1-16; P2-10 through P2-13; P3-16 through P3-20; JCS-1 through JCS-9 and JCS-18 through JCS-23	EPA 6020/ 7471
soil	pH, total organic carbon , soil moisture, water holding capacity, cation exchange capacity	28	P1-13 through P1-16; P2-10 through P2-13; P3-16 through P3-20; JCS-1 through JCS-9 and JCS-18 through JCS-23	EPA 9045; EPA 9060A; SM 2540G; SM 2540G; EPA 9081;
soil	14-day earthworm survival and biomass toxicity test; particle size analysis	7	COMP-1 through COMP-3; COMP-JC; COMP-SP; COMP-NE; COMP-BK	EPA 600/3-88-029; ASTM D 422-Atterberg Limit
soil	Lead by XRF	22 grids	Grid #s 267-288. If required, additional step out grids will be sampled	EPA 6020 (5%) of samples
soil	Lead	5	VD-1 through VD-5	EPA 6020
surface water	Fathead minnow *(P. promelus)* and water flea (*C. dubia*) toxicity test	10	P3-SW-1 and P3-SW-2, SW-1, SW-3, SW-4, SW-5, SW-18, SW-20, SW-22, SW-23	EPA-821-R-02-013 Method 1000.0
surface water	pH, water hardness	16	P3-SW-1 and P3-SW-2 SW-1 through SW-8, SW-18 through SW-23	SM 4500 H; SM 2340B
surface water	Total metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn); Dissolved metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn)	16	P3-SW-1 and P3-SW-2 SW-1 through SW-8, SW-18 through SW-23	EPA 6020/ 7470A
sediment	Metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn)	23	SD-1 through SD-23	EPA 6020/7471
sediment	Total Phenols	23	SD-1 through SD-23	EPA 8270C
sediment	*H. azteca* (28-day) and *C. dilutus* (10-day) toxicity test	11	SD-1, SD-3, SD-4, SD-5, SD-10, SD-13, SD-17, SD-18, SD-20, SD-22, SD-23	EPA/600/R-99/064 100.4 and 100.2

Sample Matrix	Analysis	Number of Samples	Sample ID (a)	Method
sediment	Interstitial pore water ammonia; Interstitial pore water pH	23	SD-1 through SD-23	EPA 350.1; SM 4500-H-B
sediment	AVS/SEM	23	SD-1 through SD-23	EPA 1629/6010/ 7471
sediment	Total organic carbon , particle size analysis, cation exchange capacity	23	SD-1 through SD-23	Walkley-Black; ASTM D 422-63; EPA 9081
ground-water	Total metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn); Dissolved metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn); total suspended solids; total phenols	11	MW-1R, MW-2 through MW-10 and MW-13	EPA 6020/7470A; SM 2540D; EPA 8270C
benthic inverte-brate	Total Metals (wet weight and dry weight), Moisture Content	23	BI-1 through BI-5, BI-8, BI-9, BI-12, BI-13, BI-15, BI-17 through BI-20, BI-22, BI-23	EPA 6020/7471/ ASTM D 2974-87

3.7 Addendum to the DERA

An addendum to the DERA will be completed to incorporate the new data and assess new receptors (i.e., Indiana bat, spotted sandpiper). As requested by U.S. EPA, the risk characterization will include fish and aquatic invertebrates in Julia Creek and the Ponds; benthic invertebrates in Julia Creek and the Ponds; and the Indiana bat, earthworms and Spotted sandpiper. The risk characterization will utilize a weight of evidence approach for the receptors that have multiple lines of evidence (i.e., earthworms, fish, pelagic and benthic invertebrates).

The DERA will include assessment of the Spotted sandpiper, American robin, and Indiana bat, an endangered species. The American robin was previously included in the 2006 assessment; however, due to the risks noted at that time, a reassessment of risks to the robin, incorporating new soil data, updated exposure pathways and toxicity reference values, will be included in the addendum. The decision to include the Indiana bat in the ERA is based on a recent observation from U.S. Fish and Wildlife Service (2007) that in recent years, "Indiana bats (Myotis sodalis) maternity colonies are utilizing forested stream corridors in the summer within a few miles of the Quemetco site. Whether or not Indiana bats are actually present at the Quemetco site in the summer is currently unknown, however, that potential exists."

The Indiana bat will be evaluated using similar methods and models that were used to assess other mammals. There are two primary differences: 1) because the Indiana bat is a threatened species, the no-observed-adverse-effect levels (NOAELs) will be used instead of the lowest-observed-adverse-effect levels (LOAELs); and 2) because the Indiana bat forages over areas from <100 to > 800 acres (Humphrey et al., 1977; Rommé et al., 1995; Menzel et al., 2005), and the entire Site is only 8 acres in size, it may be assumed that an individual Indiana bat will obtain less than 10% of its diet from the Site. At the request of U.S. EPA, risks will also be evaluated assuming the Indiana Bat obtains 100% of its diet from the Site.

Exposure parameters to be used in the assessment of Indiana bat include the following:

- Body weight = 7.1 g (US FWS, 2006)
- Food ingestion rate = 3.5 g/d (US FWS, 2006)
- Soil/sediment ingestion rate = assumed negligible, since they consume primarily flying insects (assumption used in Sample et al., 1997 for other bats)
- Water ingestion rate = 0.16 L/day (equation 21 in Sample et al., 1997)
- Diet composition = flying insects found along rivers or lakes, and in uplands (US FWS, 2006). Aquatic-based insects were eaten when riparian woodlands were the predominant available habitat (Brack, 1989). Therefore, based on input from U.S. EPA, 100% of the diet is obtained from aquatic invertebrates.
- Foraging range = varies widely from <100 to > 400 acres (Humphrey et al., 1977; Rommé et al., 1995; Menzel et al., 2005) or approximately 1 - 2 miles from their roost trees (Pruitt et al., 1995; Rommé et al., 1995). Indiana bats forage in upland forests and riparian woodlands and prefer closed canopies; they typically avoid open water, deforested creeks or open fields or pastures (Brack, 1988; Rommé et al., 1995). Risks were evaluated assuming 10% of the diet and, at the request of U.S. EPA, 100% of the diet, comes from the Site.

The equation for estimating exposure to Indiana bat is the same as the general equation for calculating intake rates for wildlife:

Intake = ((Cwater x IRwater) + (Cfood x IRfood x FI)) / BW

where:

Intake = Intake of chemical (mg/kg/day)

Cwater = Concentration of chemical in water (mg/L)

IRwater = Water ingestion rate (L/day)

Cfood = Concentration of chemical in food (mg/kg)

IRfood = Food ingestion rate (kg/day)

FI = Fraction of total daily food intake represented by food item from site (unitless)

BW = Body weight (kg)

It will be assumed that the bat forages over the areas with aquatic-based insects, i.e., along Julia Creek and the ponds. Therefore, an upper estimate of the mean concentration (the (95% upper confidence limit on the arithmetic mean) in sediments (areas where aquatic insects reside) within Julia Creek and the ponds will be used to model uptake into dietary items.

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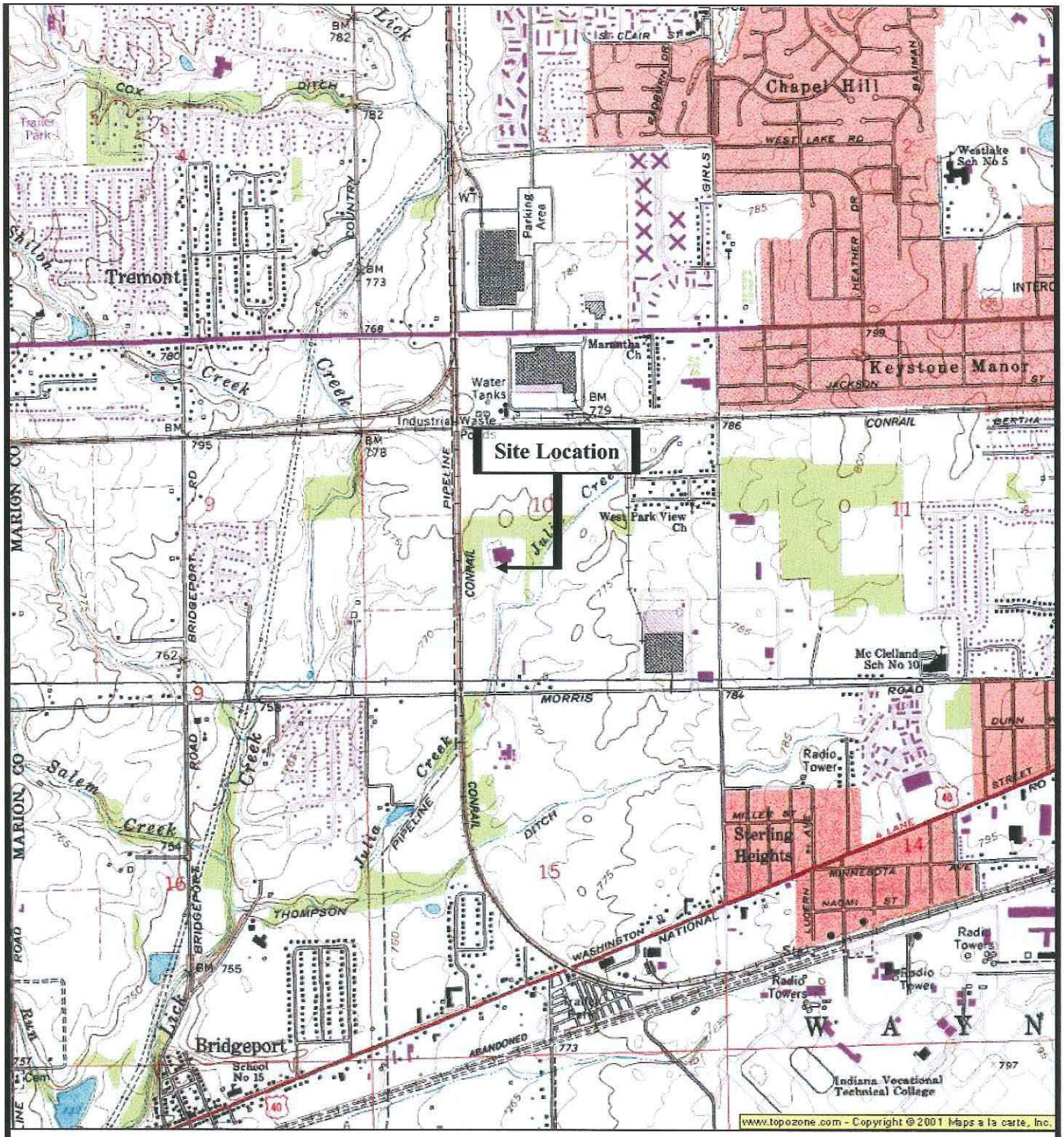
Figures

q:\client\quemetco\indiana\indianapolis, in figure 1



Reference
7.5 Minute Series Topographic Quadrangle
Clermont, Indiana
Photorevised 1984 Scale 1:25,000

Quadrangle Location

1000 0 2000 4000

Scale in Feet

WSP

2025 Gateway Place, Suite 435
San Jose, California
(408) 453-6100

Figure 1
Site Location
Location of Quemetco Facility
Indianapolis, Indiana

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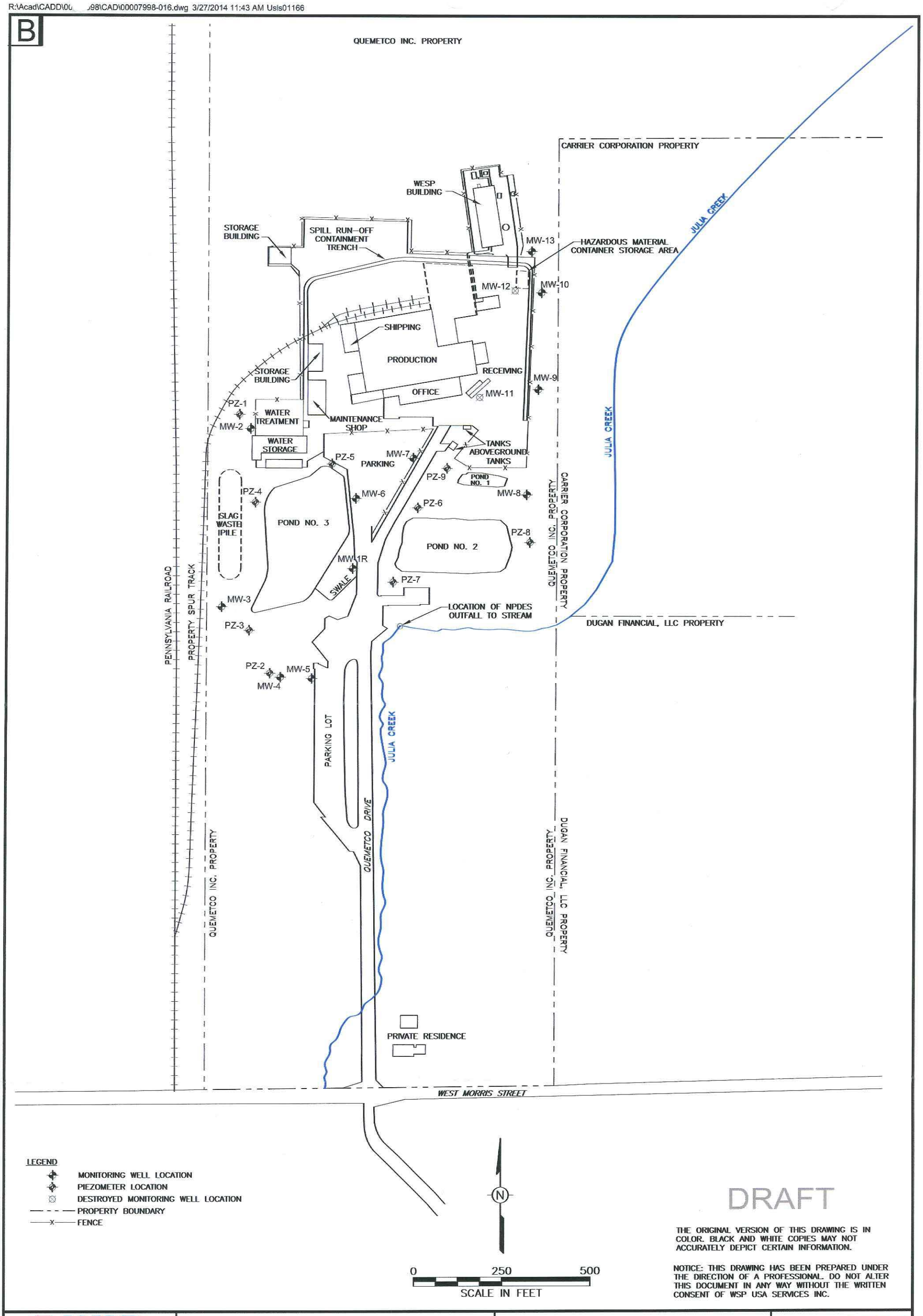

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Figure 2

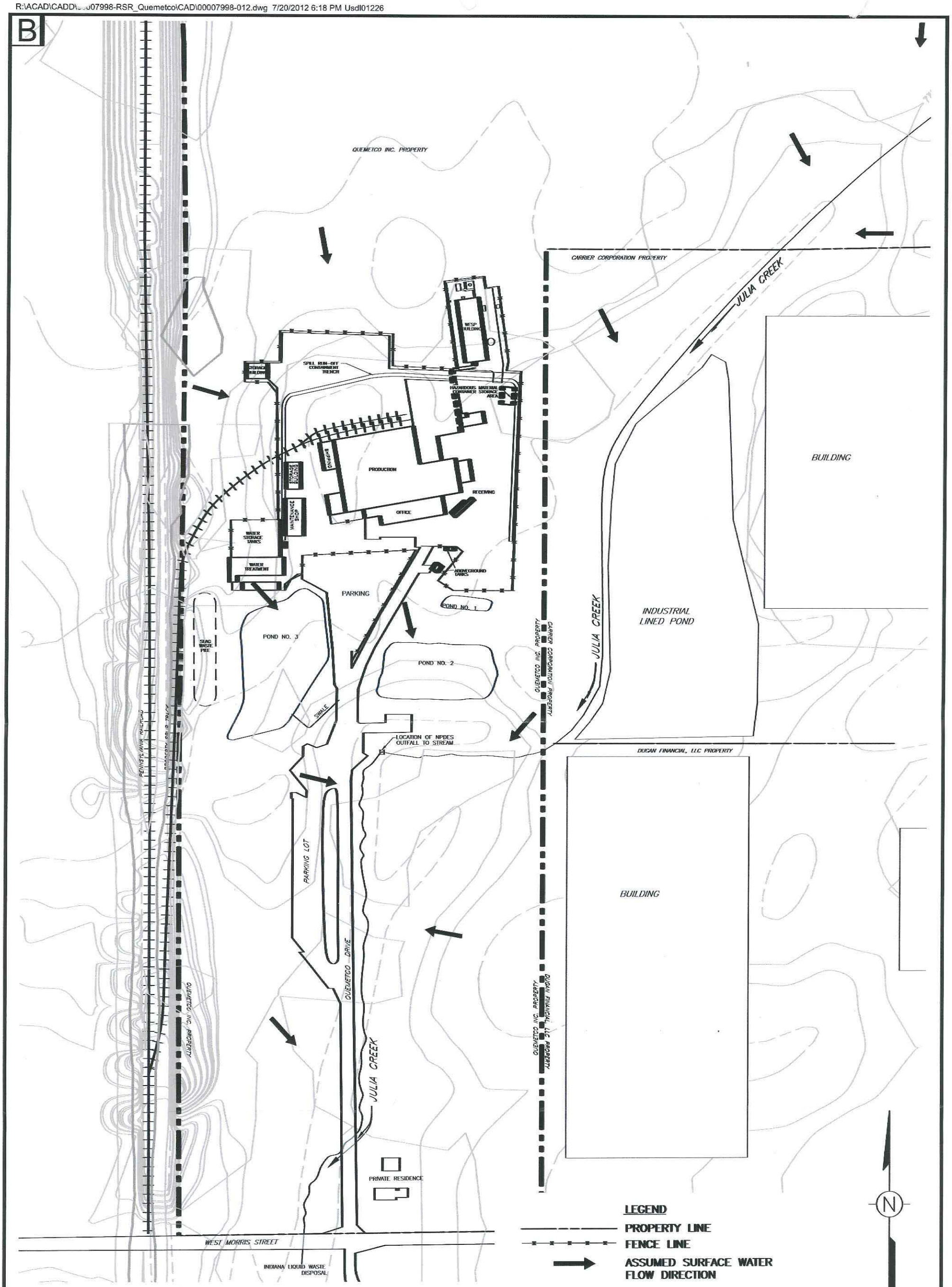
SITE PLAN

900 QUEMETCO DRIVE
INDIANAPOLIS, INDIANA

PREPARED FOR
QUEMETCO, INC.
INDIANAPOLIS, INDIANA

Drawn By:	LS 3/26/2014
Checked:	
Approved:	
DWG Name:	00007998-016

R:\ACAD\CADD\00007998-RSR_Quemetco\CAD\00007998-012.dwg 7/20/2012 6:18 PM Usdl01226



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THE ORIGINAL VERSION OF THIS DRAWING IS IN COLOR. BLACK AND WHITE COPIES MAY NOT ACCURATELY DEPICT CERTAIN INFORMATION.

REFERENCE: TOPOGRAPHIC CONTOURS DERIVED FROM USGS TOPOGRAPHIC QUAD 1:24K SERIES CLERMONT QUADRANGLE, 1998; A GOOGLEEARTH SURFACE MESH, MAY 2010

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Figure 4

SURFACE WATER FLOW

900 QUEMETCO DRIVE
INDIANAPOLIS, INDIANA

PREPARED FOR
QUEMETCO, INC.
INDIANAPOLIS, INDIANA

Drawn By:	DWL	7/06/2012
Checked:	AW	7/13/2012
Approved:	BJM	7/13/2012
DWG Name:	00007998-012	

R:\ACAD\CADD\00007998-RSR_Quemetco\CAD\00007998-011.dwg 7/20/2012 5:45 PM Usdl01226

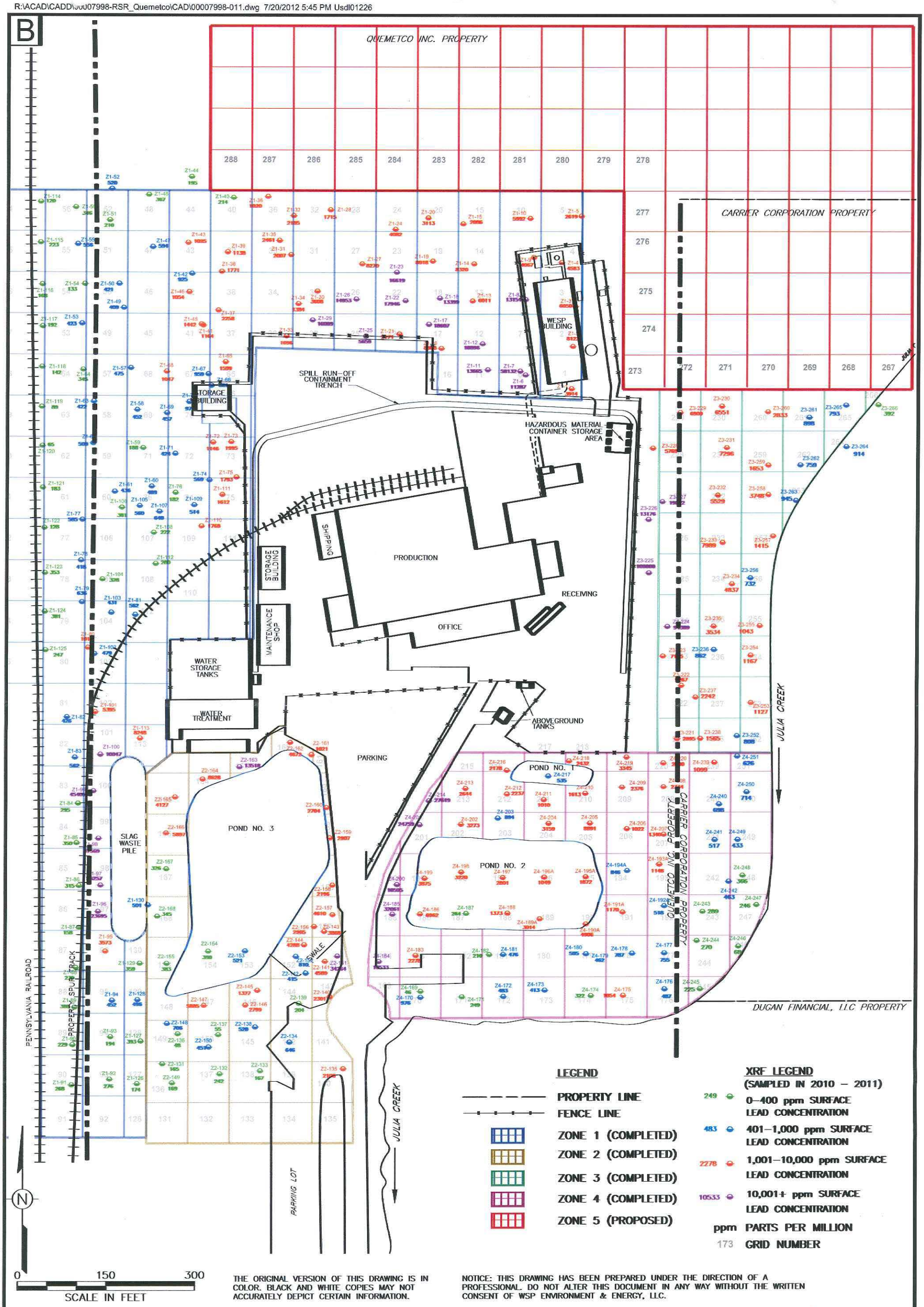

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Figure 5

PROPOSED XRF SAMPLING GRID

900 QUEMETCO DRIVE
INDIANAPOLIS, INDIANA

PREPARED FOR
QUEMETCO, INC.
INDIANAPOLIS, INDIANA

Drawn By:	[illegible]	7/20/12
Checked:	AW	7/20/12
Approved:	BJM	7/20/12
DWG Name:	00007998–011	

Table 2

Table 2-Sample Container and Preservat' 'ie for Site Characterization and DERA Sampling

Sample Matrix	Analysis	Method	Holding Time	Container type/size	Preservative	Storage Requirement	Laboratory QA/QC control limits: Method Detection Limits (MDL)	Reporting Limits (RL/PQL)	LCS/LCSD Percent Recovery	LCS/LCSD Relative Percent Difference (RPD)	MS/MSD Percent Recovery	MS/MSD Relative Percent Difference (RPD)	Laboratory	Notes
							MDL (mg/kg)	PQL (mg/kg)						
surface soil	Metals													
	Antimony	SPA 6020	6 months	4 oz jar	none	cool, <6°C	0.015	0.10	80-120	20	75-125	20	PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9408	SEE ADDITIONAL NOTES AT BOTTOM OF TABLE
	Arsenic						0.013	0.10	80-120	20	75-125	20		
	Barium						0.009	0.10	80-120	20	75-125	20		
	Cadmium						0.011	0.10	80-120	20	75-125	20		
	Chromium						0.0166	0.10	80-120	20	75-125	20		
	Copper						0.047	0.10	80-120	20	75-125	20		
	Lead						0.008	0.10	80-120	20	75-125	20		
	Nickel						0.046	0.10	80-120	20	75-125	20		
	Zinc						0.855	2.00	80-120	20	75-125	20		
	Mercury	EPA 7471A/B	28 days				0.0020	0.020	85-115	20	85-115	20		
surface soil	pH	EPA 9045	ASAP	4 oz jar	none	cool, <5°C	0.05 Std units	0.1 Std units	NA	5	NA	NA	PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9408	Water holding capacity and Particle Size subcontracted to CQM, INC.: CRC, Pace -Kansas
	Total Organic Carbon (TOC)	EPA 9060A	28 days				73.37 mg/kg	600 mg/kg	80-120	20	50-150	20		
	Soil Moisture Content	SM 2540G	NA	Three 4oz jars; 1 gallon zip lock bag			0.10%	0.10%	NA	10	NA	NA		
	Water Holding Capacity						NA	NA		NA				
	Particle size analysis	ASTM D422/ Atterberg Limit	NA											
	Cation Exchange Capacity (CEC)	EPA 9081	180 days				0.05 meq/100g	0.1 meq/100g	62-138	47	NA	NA		
soil	14-day earthworm survival and biomass toxicity test	EPA 600/3-88-029	72 hr recommended (see footnote for reference)	plastic or PTFE container; can use 1L or gal size; need 2 L	None. Fill container completely, i.e., no headspace. Chill to 4°C. Do not freeze.	Store at 4°C in the dark until used for testing.	N/A	N/A	N/A	N/A	N/A	N/A	Northwestern Aquatic Sciences 1814 Yaquina Bay Road Newport, Oregon 97365 Phone: 541-265.7225 Fax: 541-265.2799 contact@nwaquatic.com	testing 100% soil or 50% dilution series? Volume required depends on it. 100g needed ~2L
XRF soil	Lead	EPA 6020	6 months	4 oz jar	none	cool, <6°C	0.008 mg/kg	0.10 mg/kg	80-120	20	75-125	20	PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9406	soil samples collected every 10 XRF
subsurface soil	Lead	EPA 6020	6 months	4 oz jar	none	cool, <6°C	0.008 mg/kg	0.10 mg/kg	80-120	20	75-125	20	PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9407	
surface water	Fathead minnow (P. promelas) and water flea (C. dubia) (chronic) toxicity test	EPA-821-R-02-013 Method 1000.0	48 hr ice as soon as possible after collection	5 gal cubitainer of a grab sample	None. Fill container completely, i.e., no headspace. Chill to 4°C. Do not freeze.	Store at 4°C in the dark until used for testing.	N/A	N/A	N/A	N/A	N/A	N/A	Northwestern Aquatic Sciences 1814 Yaquina Bay Road Newport, Oregon 97365 Phone: 541-265.7225 Fax: 541-265.2799 contact@nwaquatic.com	testing 100% surface water or 50% dilution series? 5-gal will cover dilution series. Can only run 5 or 6 samples at one time if doing dilution series.
surface water	pH	SM 4500-H+	ASAP	2 plastic 250 ml	1- none; 1 HNO3 <2pH	cool, <6°C	0.01 Std Units	0.1 Std Units	NA	5	NA	NA	PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9407	
	water hardness	SM 2340B	6 months	2 plastic 250 ml	1- none; 1 HNO3 <2pH		150 ug/l	2000 ug/l	NA	20	NA	NA		
surface water	Metals (Total and Dissolved)	EPA 6020	6 months	2 plastic 250 ml	pH<2; HNO3	cool, <6°C	MDL (ug/L)	PQL (ug/L)					PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9408	It is assumed that water samples will be field filtered for dissolved metals
	Antimony						0.115	1	80-120	20	75-125	20		
	Arsenic						0.193	1	80-120	20	75-125	20		
	Barium						0.199	1	80-120	20	75-125	20		
	Cadmium						0.130	1	80-120	20	75-125	20		
	Chromium						0.1156	1	80-120	20	75-125	20		
	Copper						0.381	1	80-120	20	75-125	20		
	Lead						0.061	1	80-120	20	75-125	20		
	Nickel						0.155	1	80-120	20	75-125	20		
	Zinc						4.069	10	80-120	20	75-125	20		
	Mercury	EPA 7470A	28 days				0.100	0.200	85-115	20	85-115	20		
sediment	Metals	EPA 6020	6 months	4 oz glass jar	none	cool, <6°C	MDL (mg/kg)	PQL (mg/kg)					PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9407	
	Antimony						0.015	0.10	80-120	20	75-125	20		
	Arsenic						0.013	0.10	80-120	20	75-125	20		
	Barium						0.009	0.10	80-120	20	75-125	20		
	Cadmium						0.011	0.10	80-120	20	75-125	20		
	Chromium						0.0166	0.10	80-120	20	75-125	20		
	Copper						0.047	0.10	80-120	20	75-125	20		
	Lead						0.008	0.10	80-120	20	75-125	20		
	Nickel						0.046	0.10	80-120	20	75-125	20		
	Zinc						0.855	2.00	80-120	20	75-125	20		
	Mercury	EPA 7471A/B	28 days				0.0050	0.020	85-115	20	85-115	20		
sediment	Phenols Analyte	EPA 8270C	7 days extraction/ 40 days analysis	4 oz glass jar	none	cool, <6°C	True MDL (ug/kg)	PQL (ug/kg)					PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9407	
	2,3,4,6-Tetrachlorophenol						88.52	333	10-150	20		40		
	2,4,5-Trichlorophenol						35.47	167	66-130	20		21		
	2,4,6-Trichlorophenol						42.46	167	64-130	20		33		
	2,4-Dichlorophenol						27.8	167	66-130	20		31		
	2,4-Dimethylphenol						31.83	167	66-130	20		29		
	2,4-Dinitrophenol						67.69	333	11-130	20		40		
	2-Chlorophenol						30.08	167	58-130	20		31		
	2-Methylphenol(o-Cresol)						32.59	167	65-130	20		29		
	2-Nitrophenol						32.59	167	63-130	20		41		
	3&4-Methylphenol(m&p Cresol)						24.38	167	63-130	20		29		
	4,6-Dinitro-2-methylphenol						17.52	167	35-130	20		50		
	4-Chloro-3-methylphenol						32.35	167	66-130	20		31		
	4-Nitrophenol						44.78	167	51-130	20		47		
	Cresols (Total)						56.97	333	10-159	20		40		
	Pentachlorophenol						27.54	167	45-130	20		50		
	Phenol						52.5	167	62-130	20		30		
sediment	*H. azteca* (28-day survival and growth) and *C. dilutus* (10-day) toxicity tests	EPA/600/R-99/064 100.4 and 100.1	Normally 2 weeks; possibly up to 6 wks stored under nitrogen	plastic or PTFE container, can use 1L or gal size; need 5 L (see footnote for reference)	None. Fill container completely, i.e., no headspace. Chill to 4°C. Do not freeze.	Store at 4°C in the dark until used for testing.	N/A	N/A	N/A	N/A	N/A	N/A	Northwestern Aquatic Sciences 1814 Yaquina Bay Road Newport, Oregon 97365 Phone: 541-265.7225 Fax: 541-265.2799 contact@nwaquatic.com	5L needed to do both a 10-d midge and 28-d Hyalella; allows for potential retest
sediment	Interstitial pore water Ammonia	EPA 350.1	28 days	Sediment -5 gallon covered bucket. Pore Water 250 ml plastic	H2SO4 <2pH	cool, 4°C	0.250 mg/L	0.500 mg/L	90-110	20	90-110	20	PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9407 Pore Water Generation and pH on pore water, RTI, Livonia, MI.	
	Interstitial pore water pH	SM 4500-H+B	ASAP		none		0.250 mg/L	0.500 mg/L	NA	5	NA	NA		
sediment	AVS/SEM (Acid Volatile Sulphide/Simultaneously Extracted Metals)			4 oz glass jar, no headspace	none	cool, <6°C	True MDL (umoles/g)	PQL (umoles/g)					PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9407	
	AVS	EPA 1629	14 days for AVS/SEM extraction, subsequent 14 days for AVS				0.00784605	0.05381000	50-150	30	50-150	30		
	Metals	EPA 6010	6 months				MDL (umoles/g)	PQL (umoles/g)						
	Antimony						0.00049440	0.00410840	50-150	30	50-150	30		
	Arsenic						0.00223400	0.00667360	50-150	30	50-150	30		
	Cadmium						0.00022180	0.00111210	50-150	30	50-150	30		
	Chromium						0.00073300	0.00240546	50-150	30	50-150	30		
	Copper						0.00132830	0.00393420	50-150	30	50-150	30		
	Lead						0.00048500	0.00420660	50-150	30	50-150	30		
	Nickel						0.00058810	0.00435800	50-150	30	50-150	30		
	Zinc						0.00220440	0.01255200	50-150	30	50-150	30		
	Silver						0.00032280	0.00231360	50-150	30	50-150	30		
	Mercury	EPA 7471A/B	28 days				0.00001250	0.00002400	50-150	30	50-150	30		
sediment	Total Organic Carbon (TOC)	Walkley-Black	28 days	4 oz glass jar	none	cool, <6°C	115.1 mg/kg	400 mg/kg	80-120	20	80-120	29	PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9407	
	Particle size analysis	ASTM D 422-63	NA	Two 4 oz glass jar; 1 gallon ziplock bag		none	NA	NA	NA	NA	NA	NA	CQM Inc. Green Bay, WI	
	Cation Exchange Capacity (CEC)	EPA 9081	180 days			cool, <6°C	0.5 meq/100g	0.1 meq/100g	62-138	47	NA	NA	Pace -Kansas	
ground-water	Total suspended solids (TSS)	SM 2540D	7 Days	1 Liter plastic	none	cool, <6°C not freeze	0.15 mg/L	10 mg/L	80-120	10	80-120	20	PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9407	It is assumed that water samples will be field filtered for dissolved metals. 7 Rec.Phenols. Pace Kansas
	Phenols Analyte	EPA 8270C	7 days extraction/ 40 days analysis	1 Liter glass	none		True MDL (ug/L)	PQL (ug/L)						
	2,3,4,6-Tetrachlorophenol						4.51	5	70-130	20	70-130	20		
	2,4,5-Trichlorophenol						0.76	5	70-130	20	65-130	22		
	2,4,6-Trichlorophenol						1.05	5	70-130	20	64-130	20		
	2,4-Dichlorophenol						1.19	5	69-130	23	69-130	27		
	2,4-Dimethylphenol						0.93	5	34-130	33	10-135	41		
	2,4-Dinitrophenol						0.86	10	38-130	38	12-161	46		
	2-Chlorophenol						1.03	5	38-130	27	47-139	41		
	2-Methylphenol(o-Cresol)						0.96	5	46-130	28	33-130	39		
	2-Nitrophenol						0.85	5	70-130	20	33-130	21		
	3&4-Methylphenol(m&p Cresol)						1.28	5	43-130	29	35-130	39		
	4,6-Dinitro-2-methylphenol						0.62	5	53-130	20	15-130	33		
	4-Chloro-3-methylphenol						1.43	5	61-130	27	51-130	33		
	4-Nitrophenol						2.25	10	18-130	32	10-130	25		
	Cresols (Total)						2.24	5	10-159	20	10-159	20		
	Pentachlorophenol						0.75	10	54-130	29	10-149	32		
	Phenol						0.54	5	28-130	20	23-130	33		
	Metals (Total and Dissolved)	EPA 6020	6 months	Two 250 ml. plastic	HNO3 <2pH		MDL (ug/L)	PQL (ug/L)						
	Antimony						0.115	1.00	80-120	20	75-125	20		
	Arsenic						0.193	1.00	80-120	20	75-125	20		
	Barium						0.199	1.00	80-120	20	75-125	20		
	Cadmium						0.130	1.00	80-120	20	75-125	20		
	Chromium						0.1156	1.00	80-120	20	75-125	20		
	Copper						0.381	1.00	80-120	20	75-125	20		
	Lead						0.061	1.00	80-120	20	75-125	20		
	Nickel						0.155	1.00	80-120	20	75-125	20		
	Zinc						4.069	10.00	80-120	20	75-125	20		
	Mercury	EPA 7470A	28 days				0.100	0.200	85-115	20	85-115	20		
Benthic Invertebrates	Metals	EPA 6020 in tissues	365 days	Ziplock type bags, double bagged with labels on both the interior and exterior bags.	None	Freeze and shipped on dry ice. Need at least 5 grams	MDL (mg/kg)	PQL (mg/kg)					PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9408	Sample mass needed for metals is 5 grams
	Antimony						0.006	0.10	80-120	20	75-125	20		
	Arsenic						0.014	0.10	80-120	20	75-125	20		
	Barium						0.058	0.10	80-120	20	75-125	20		
	Cadmium						0.011	0.10	80-120	20	75-125	20		
	Chromium						0.0388	0.10	80-120	20	75-125	20		
	Copper						0.064	0.50	80-120	20	75-125	20		
	Lead						0.049	0.10	80-120	20	75-125	20		
	Nickel						0.018	0.10	80-120	20	75-125	20		
	Zinc						0.890	2.00	80-120	20	75-125	20		
	Mercury	EPA 7471 A/B					0.00134	0.0600	85-115	20	85-115	20		
	Soil Moisture Content	ASTM D2974-87	28 days	100g glass with tight fitting lid		0°C to 6°C	0.10%	0.10%	NA	5	NA	NA		
	Wet Weight	NA	NA	NA		NA	NA	NA	NA	NA	NA	NA		
	Dry Weight	NA					NA	NA	NA	NA	NA	NA		

Pace Notes
Laboratory performing the work is Pace Green Bay unless otherwise noted
Method Detection Limits and Reporting Limits are periodically updated and subject to change without notice.
Pace Standard QC Level IV and Pace Standard excel EDD will be provided, except for Particle Size. Water Holding Capacity, and pore water pH.
Particle Size and Water Holding Capacity will be done by CQM, Inc. Green Bay, WI
Water Holding Capacity defined as available water capacity of soil to determine efficiency of soil in crop production, analysis required include Sieve, Hydrometer, and Atterberg Limit
Particle Size sections both sieve and hydrometer are required.
Pore Water Extraction will be done by RTI, Livonia, MI sediment to be shipped directly to RTI via ground shipment by WSP. Any overlying water on the sample when received will be considered pore water and incorporated.
Pore Water pH will be done by RTI, their QC Level IV report will be appended to Pace's. RTPs results will not be included in the EDD
AVS/SEM requires 3 week prior notice or meeting hold times is not guaranteed. All samples for this analysis must be submitted at the same time. AVS/SEM metals = Sb, As, Cd, Cr, Cu, Pb, Hg, Zn. Ba will not be included.
It is assumed that water samples will be field filtered for dissolved metals.

Appendix A – Sampling and Analysis Plan

Appendix A—Sampling and Analysis Plan to Complete Site Characterization and Address Outstanding DERA Issues

Quemetco, Inc., 900 Quemetco Drive, Indianapolis, Indiana
US EPA Identification Number: IND 000 199 653

Revision 2

3/28/2014

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Table A2 – Sample Container and Preservative Guide for Site Characterization and DERA Sampling (see blue divider sheet in back of report)

Attachments

Attachment A – WSP Standard Operating Procedures

Attachment B – Field Sampling Equipment Manufacturer's Manuals

Attachment C – Groundwater Purging and Sampling Log Form

1 Introduction

This document presents the Sampling and Analysis Plan (SAP) for completion of Site Characterization and Addressing Outstanding Detailed Ecological Risk Assessment (DERA) issues at the Quemetco, Inc. (Quemetco) Facility located at 900 Quemetco Drive, in Indianapolis, Marion County, Indiana ("Facility" or "Site") (Figures A1 and A2).

In accordance with the Workplan to Complete Site Characterization and Address Outstanding DERA Issues (Workplan), dated March 28, 2014, additional Site characterization sampling activities will include the following:

- X-Ray Fluorescence (XRF) soil sampling and confirmation soil sampling north and northeast of the plant to complete lateral delineation of lead in soil.
- Soil sampling to delineated the vertical extent of lead in soil using a Geoprobe sampling rig.
- Groundwater sampling from existing monitoring wells MW-1R, MW-2 though MW-10, and MW-13.

The groundwater monitoring wells are shown on Figure A2, the XRF sampling locations are shown in Figure A3, and the Geoprobe boring locations are shown on Figure A4.

The DERA field work will involve:

- Collect composite soil samples from around the slag pile area, on the northeast side of the plant, along Julia Creek, from Pond Nos. 1, 2 and 3, and in a background area to conduct earthworm toxicity testing.
- Soil matrix sampling around Pond Nos. 1, 2, and 3, and in several locations along Julia Creek for metals and chemistry testing.
- Surface water samples will be collected from Pond No. 3 and Julia Creek for chemistry and toxicity testing using *P. promelas* and *C. dubia.*
- Sediment samples will be collected from Pond Nos. 1, 2, and 3 and Julia Creek for chemistry, physical characterization, and toxicity testing including *Hyalella azteca* 28-day toxicity test and 10-day *C. dilutus* test. Additionally, sediment data will be used to assess the risk to the Indiana bat.
- Benthic invertebrate sampling in Julia Creek and Pond Nos. 1, 2, and 3 for tissue analysis with results used in the risk characterization utilizing a weight of evidence approach for the receptors.

The proposed DERA sampling locations are presented in Figure A5.

A summary of the proposed Site Characterization and DERA samples and the corresponding analyses is provided in Table A1.

Table A1- Site Characterization and DERA Sample Collection, Analysis and Laboratory Assignment

Sample Matrix	Analysis	Number of Samples	Sample ID (a)	Method	Laboratory
soil	Metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn)	28	P1-13 through P1-16; P2-10 through P2-13; P3-16 through P3-20; JCS-1 through JCS-9 and JCS-18 through JCS-23	EPA 6020/ 7471	Pace Analytical
soil	pH, total organic carbon , soil moisture, water holding capacity, cation exchange capacity	28	P1-13 through P1-16; P2-10 through P2-13; P3-16 through P3-20; JCS-1 through JCS-9 and JCS-18 through JCS-23	EPA 9045; EPA 9060A; SM 2540G; SM 2540G; EPA 9081;	Pace Analytical; CQM for water holding capacity
soil	14-day earthworm survival and biomass toxicity test; particle size analysis	7	COMP-1 through COMP-3; COMP-JC; COMP-SP; COMP-NE; COMP-BK	EPA 600/3-88-029; ASTM D 422-Atterberg Limit	NAS; CQM
soil	Lead by XRF	22 grids	Grid #s 267-288. If required, additional step out grids will be sampled	EPA 6020 (5% of samples)	WSP will conduct in field; Pace Analytical will conduct laboratory verification
soil	Lead	5	VD-1 through VD-5	EPA 6020	Pace Analytical
surface water	Fathead minnow *(P. promelus)* and water flea (*C. dubia*) toxicity test	10	P3-SW-1 and P3-SW-2, SW-1, SW-3, SW-4, SW-5, SW-18, SW-20, SW-22, SW-23	EPA-821-R-02-013 Method 1000.0	NAS
surface water	pH, water hardness	16	P3-SW-1 and P3-SW-2 SW-1 through SW-8 and SW-18 through SW-23	SM 4500 H; SM 2340B	Pace Analytical
surface water	Total metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn); Dissolved metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn)	16	P3-SW-1 and P3-SW-2 SW-1 through SW-8 and SW-18 through SW-23	EPA 6020/ 7470 A	Pace Analytical
sediment	Metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn)	23	SD-1 through SD-23	EPA 6020/7471	Pace Analytical
sediment	Total Phenols	23	SD-1 through SD-23	EPA 8270C	Pace Analytical

Sample Matrix	Analysis	Number of Samples	Sample ID (a)	Method	Laboratory
sediment	*H. azteca* (28-day toxicity); *C. dilutus* (10-day toxicity)	11	SD-1, SD-3, SD-4, SD-5, SD-10, SD-13, SD-17, SD-18, SD-20, SD-22, SD-23	EPA/600/R-99/064 100.4 and 100.2	NAS
sediment	Interstitial pore water ammonia; Interstitial pore water pH	23	SD-1 through SD-23	EPA 350.1; SM 4500-H-B	Pace Analytical; RTI (Pore water generation and pore water pH)
sediment	AVS/SEM	23	SD-1 through SD-23	EPA 1629/6010/ 7471	Pace Analytical
sediment	Total organic carbon , particle size analysis, cation exchange capacity	23	SD-1 through SD-23	Walkley-Black; ASTM D 422-63; EPA 9081	Pace Analytical; CQM for particle size analysis
ground-water	Total metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn); dissolved metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn); total suspended solids; total phenols	11	MW-1R, MW-2 through MW-10 and MW-13	EPA 6020/ 7470A; SM 2540D; EPA 8270C	Pace Analytical
benthic inverte-brate tissue	Total Metals (wet weight and dry weight), Moisture Content	16	BI-1 through BI-5, BI-8, BI-9, BI-12, BI-13, BI-15, BI-17 through BI-20, BI-22, BI-23	EPA 6020/7471/ ASTM D 2974-87	Pace Analytical

Four laboratories will be used to complete the analytical work for this project. Northwestern Aquatic Sciences (NAS) and Pace Analytical Laboratories (Pace Analytical) in Indianapolis, Indiana will perform surface water, soil, groundwater and sediment analyses. The benthic invertebrate analyses for metals will be conducted by Pace Analytical's Green Bay, Wisconsin lab. RTI Laboratories in Livonia, Michigan will perform the sediment pore water extraction and pH analysis. CQM of Green Bay, Wisconsin will perform the particle size and water holding capacity analyses. RTI and Pace Analytical are National Environmental Laboratory Accreditation Program (NELAP) certified laboratories. NAS is a state certified laboratory. CQM is certified by the Wisconsin Department of Transportation.

All field activities will be conducted in accordance with WSP's Standard Operating Procedures (SOPs), which will support the project objectives by ensuring that reliable data is produced from the sample collection activities. The SOPs are presented in Attachment A and address all aspects of sample collection including field documentation; sampling procedures for soil, sediment, surface water; equipment decontamination; field quality assurance/quality control (QA/QC) samples; and managing investigation-derived waste.

The field activities will also be conducted in accordance with the Health and Safety Plan (HASP) for this project (Appendix C of the Workplan).

2 Site Characterization Field Activities

This section presents the sampling plan designed to further characterize the lateral and vertical extent of lead in soil across the Site and establish current groundwater conditions. A summary of the analytical program for soil and groundwater samples is included on Table A1 above. The approach to soil characterization is to supplement previous sampling events, including the most recent XRF soil investigation, and fill in data gaps where lead impacts are unknown.

2.1 Decision Statements

Soil samples will be collected from various locations throughout the Site. These samples will be used to further delineate the lateral and vertical extent of contamination at the Site. The target action level for lead is 400 milligrams/kilograms (mg/kg or parts per million (ppm)), which is the EPA Region 9 residential direct-contact action level. Sampling will continue laterally until lead concentrations as determined by the XRF are below 400 mg/kg. The XRF results are not being compared to the 2003 Region 5 ecological screening levels (ESLs) of 0.05 mg/kg due to the fact that this value is below the background concentrations of lead in soil and approximates the laboratory reporting limit for lead. Therefore, with the approval of EPA, an ecological-based Action Level has been developed for lead. In a memo from Intrinsik Environmental Sciences to EPA, dated February 14, 2014, it was calculated determined that the overall Ecological Action Level for lead is 130 mg/kg (Intrinsik Environmental, 2014). Furthermore, it was agreed upon that the Ecological Action Level will not be used to screen the data and that any proposed remediation criteria will likely be based on the ecological risk assessment and not this Ecological Action Level. The XRF data will be compared to the laboratory analysis of total lead. Laboratory samples will be collected from homogenized soil 1-6 inches below ground surface (bgs) and the number of samples collected will consist of five percent (5%) of the total XRF measurements collected. The samples will be analyzed in the laboratory using inductively coupled plasma-mass spectrometry (ICP-MS) via EPA Methods 6020. Correlation between XRF sampling and laboratory data will be accomplished by calculating the relative percent difference (RPD) between the two results (XRF and laboratory data). No pass/fail criteria will be established for this portion of the project. Use of the newly generated XRF data is as a screening tool to broadly delineate the lead contamination. A difference in moisture content is anticipated between the soil tested in the field using the XRF compared to the samples being tested in the laboratory recorded in dry weight, WSP does not expect the field readings and the laboratory results to match precisely. Furthermore, the XRF is calibrated to a 1,345 ppm lead standard and operates best within the lower range of lead concentrations. It was observed from the previous XRF investigation that at high concentrations (greater than 1,295 ppm) the results varied more greatly from the fixed-laboratory generated results.

2.2 XRF Sampling Procedures

In order to complete the lateral delineation of lead in soil to the north and northeast of the plant, WSP will use the XRF sampling strategy and methodologies implemented in the 2010/2011 XRF soil investigations. The procedures that follow were taken directly from WSP's XRF Sampling and Analyses Plan dated February 19, 2010. These procedures followed the US EPA's approach presented in their letter dated October 8, 2009. The sampling strategy involves creating a grid composed of 70 foot by 70 foot squares within the target areas of the Site and collecting XRF surficial soil samples (0 to 1 inch

below ground surface (bgs)) from within each grid square. XRF readings are measured in deeper soils (1 to 6 inches bgs) in alternate grid squares, which results in half of the gridded area being characterized to a depth of 6 inches bgs. Also, in compliance with the EPA's recommendation, 5% of the samples undergoing XRF measurements will be sent to a fixed laboratory for verification analysis. The laboratory verification samples will only be collected from the soils at 1-6 inches bgs and encountered in alternate grid squares during XRF screening. The laboratory verification analysis for lead will be conducted using a SW-846 guidance method (e.g. EPA Method 6020).

The proposed XRF sampling locations are represented on Figure A3 by the empty grids (Zone 5) north and northeast of the plant. XRF readings/samples will be taken first in the row of grid squares nearest the plant labeled as 267 through 288 on Figure A3. If an XRF reading exceeds 400 mg/kg lead in a grid square, then the investigation will extend into select grid squares immediately adjacent to the grid square where the XRF reading was above 400 mg/kg. Thus, additional XRF readings may only be collected in specific squares and not necessarily an entire row, as applicable. XRF sampling will continue laterally until the lead concentrations have been delineated to below 400 mg/kg.

XRF may extend to the Quemetco property line to the north and northeast if step out readings are required per the strategy described above. Quemetco has obtained an access agreement from Carrier Corporation to continue sampling on their property (Figure A3). The sample grids on the Carrier Corporation property will be extended as far as necessary but not beneath any buildings or structures.

Each sample location will be recorded using a global positioning system (GPS) device. A map depicting all sample locations and surrounding areas will be provided with the final report. WSP will notify U.S. EPA at least 48 hours before any scheduled fieldwork.

2.2.1 XRF Surficial Zone Soil Sampling

XRF readings on surficial soil (0 to 1 inch bgs) will be collected in accordance with SW-846, Guidance Method 6200, section 11.3 (Feb. 2007). WSP SOP # 31 for XRF sampling is included in Attachment A. Additionally, the XRF manufacturer's manual is included in Attachment B.

Surficial zone soil sampling will be conducted as follows:

- Clear the sample area of debris, rocks, pebbles, and organic matter.
- The soil surface must be smooth to provide good contact with the soil sampling protection plate of the XRF device. If necessary, the surface will be leveled with a stainless-steel trowel or spoon.
- The surface will be tamped to increase soil density, based on the SW-846 protocol; this process will allow better repeatability and ensure that the sample is representative.
- The XRF does not perform well for saturated soils; therefore, WSP will not conduct XRF sampling unless there has been no precipitation at the Site for at least 48 hours. Additionally, WSP will not conduct XRF sampling during a storm event. WSP field representatives will ensure soil is not saturated prior to XRF analysis by conducting soil moisture measurements at each sampling location using a soil moisture meter (e.g., General DSMM500 Soil Moisture Meter). If soil moisture content is greater than 20%, a new location within the grid will be selected and the soil moisture content will be measured. This procedure will continue until a location within the grid is identified as having a soil moisture content of less than 20%. If a viable sampling location is not available within the grid, a soil sample from within the grid will be collected for fixed-laboratory analysis.

- The field lead analysis will be performed in accordance with the manufacturer's manual for operation of XRF instrumentation (Attachment B). The XRF device (along with a soil sampling protection plate) will be placed directly on the sampling location for a minimum of 60 nominal seconds (i.e., 60 seconds counted by the XRF device). Based on manufacturer's specifications, this method of analysis detects lead in the upper few millimeters of soil in contact with the soil protection plate.

The following XRF surficial zone soil sampling information will be recorded in a field notebook:

- sample zone location
- sample GPS coordinates
- sample identification numbers
- date and time
- sample depth
- field lead concentration detected
- soil moisture reading
- description of any visible evidence of soil contamination (i.e., odor, staining)

2.2.2 XRF Subsurface Zone Soil Sampling

XRF readings of subsurface soil samples (1 to 6 inches bgs) for lead analysis will be collected in accordance with SW-846, Guidance Method 6200, Section 11.4 (Feb. 2007). Under Section 11.4, ex-situ soil samples should be homogenized, dried, and ground before XRF analysis. WSP contacted EPA requesting a variance for the homogenization and drying techniques described in Sections 11.4 and 11.5, respectively. With EPA's approval, WSP will not be utilizing fluorescent dye and ultraviolet light for homogenization or the drying technique using an oven with two to four hours of drying time.

All subsurface zone soil samples will be collected at the same locations as the surficial zone soil sampling locations; however, the subsurface zone soil samples will be collected at a sample interval depth of 1 to 6 inches bgs. Subsurface soil samples for lead analysis will be collected with a single hand trowel or stainless steel spoon. The trowel and/or spoon will be decontaminated before use and between locations. Decontamination procedures are described in Section 4.3, page 23, and included as WSP SOP #19 in Attachment A. Subsurface zone soil sampling will be conducted in accordance with WSP SOP #31 for XRF sampling included in Attachment A. Additionally, the XRF manufacturer's manual is included in Attachment B.

Subsurface zone soil sampling procedures are outlined in WSP SOP #2 (Attachment A). The sampling procedures will include the following:

- Using the trowel, loosen and homogenize the soil in a small area until a depth of approximately six inches is reached.
- Lift a portion of the soil out with the blade and place in a new plastic bag.
- Break down large clumps of soil within the plastic bag while further homogenizing the sample.
- As stated above, the XRF instrument does not perform well for saturated soils. WSP field personnel will conduct soil moisture content measurements in accordance with SOP #31 and proceed as specified above.

- The field lead analysis will be performed in accordance with the manufacturer's manual for operation of XRF instrumentation. The XRF device will be placed directly on the plastic bags for a minimum of 60 nominal seconds according to manufacturer's specifications.
- For samples requiring laboratory analysis, transfer the contents of the plastic bag to a single 4-ounce laboratory provided container (i.e., glass jar) using the sampling trowel or spoon.
- Label and place the sample in a cooler containing ice.
- Decontaminate sampling equipment to be used at subsequent sampling locations.

The following subsurface zone soil sampling information will be recorded in a field notebook:

- sample zone location
- sample GPS coordinates
- sample identification numbers
- date and time of sampling
- sample depth
- field lead concentration detected
- soil moisture reading
- description of any visible evidence of soil contamination (i.e., odor, staining)

Upon completion of all sampling activities, all sampling-holes will be backfilled with soil cuttings and tamped down.

2.2.3 Analytical Program

Five percent (5%) of the total XRF measurements will be analyzed at a fixed laboratory for total lead according to the SW-846 guidance method using ICP-MS (6020). As described above, only subsurface zone soil samples will be collected for fixed-laboratory analysis. The samples will be analyzed from the homogenized field sample. Laboratory analyses will be performed by Pace Analytical, who will also provide the jars and coolers used to preserve and transport the samples. The specific number and types of sample containers required for lead analysis are included in Table A2 and in SOP #2. Chain-of-custody documentation will be maintained in accordance with WSP SOP #20 and described in Section 4.1, page 22. The analytical report will include a Level IV data package.

2.2.4 Quality Assurance/Quality Control

For quality assurance/quality control (QA/QC) purposes, one duplicate and one matrix spike/matrix spike duplicate (MS/MSD) sample will be collected and analyzed for lead. Additionally, one equipment blank sample for decontamination of the spoon/trowel and bowls will be collected and analyzed for lead.

A summary of QA/QC sample collection and analysis is provided in the QAPP (Appendix B of the Workplan). The field QA/QC samples will be collected in accordance with SOP #21.

2.3 Vertical Delineation of Lead

In order to delineate the vertical extent of lead contamination in soil, WSP will advance five (5) soil borings in areas of the Site that have been identified as requiring vertical characterization and will supplement previous vertical soil sampling data from the Site. The soil samples will be collected at a depth of 8 feet bgs. Each boring location will be cleared before drilling using a private utility locating service. The soil boring locations are presented on Figure A4 and detailed procedures for soil sampling using direct push equipment is presented in WSP SOP #24 (Attachment A).

2.3.1 Soil Sampling using Direct Push Drilling

Five borings (VD-1 through VD-5) will be drilled in the locations shown on Figure A4 using a direct push drilling rig (Geoprobe® or equivalent). The drilling rig will be used to advance 3.5-inch outside diameter dual-tube drill rods equipped with Lexan or acetate (or equivalent) sleeves. Dual tube sampling equipment will be used as the outer rods remain in the ground to prevent soil sloughing when the sampler (inner tube) is removed. To collect a continuous soil core, the sampler will be pushed in 4-foot intervals. Soil samples for laboratory analysis will be collected from each boring at depths intervals of 1 to 1.5 feet bgs; 3 to 3.5 feet bgs; and 7 to 7.5 feet bgs.

The sleeve(s) from the desired target depth(s) will be covered with Teflon tape, capped, and labeled. The samples will be handled according to procedures described in Section 4.1, page 22, and included in WSP SOPs # 2 and 20 (Attachment A). Upon completion of the soil collection procedure, the borehole will be backfilled with hydrated bentonite granules.

WSP will notify U.S. EPA at least 48 hours before any scheduled fieldwork. The GPS coordinates of the each boring will be obtained using a portable GPS unit. A map depicting all sample locations and surrounding areas will be provided with the final report.

2.3.2 Analytical Program

The 4-ounce laboratory supplied glass jars containing the soil sample will be placed in an iced cooler for transport to the laboratory. Soil samples will be analyzed by Pace Analytical for lead by EPA method 6020 (Table A2).

The analytical report will be presented as a Level IV data package.

2.3.3 Quality Assurance/Quality Control

For QA/QC purposes, one duplicate and one MS/MSD sample will be collected and analyzed for lead. Additionally, one equipment blank sample for decontamination of the drill rig equipment will be collected and analyzed for lead.

A summary of QA/QC sample collection and analysis is provided in the QAPP (Appendix B of the Workplan). The field QA/QC samples will be collected in accordance with SOP #21.

2.4 Groundwater Sampling Methodology

The current groundwater monitoring network at the Site consists of 11 monitoring wells (MW-1R, MW-2 through MW-10 and MW-13) and nine piezometers (PZ-1 through PZ-9) (Figure A2).

A one-time sampling event is proposed to assess the current groundwater flow direction and groundwater quality. The work will involve development of each of the wells and piezometers, measurement of water levels in all 20 wells and piezometers, and sampling the 11 monitoring wells. Also, each of the wells and piezometers will be re-surveyed for vertical elevation. Groundwater sampling procedures are described below and presented in SOP#3 included in Attachment A.

2.4.1 Well Development

As indicated in the Workplan, monitoring wells MW-1R, MW-2 through MW-10, MW-13, and piezometers PZ-1 through PZ-9 will be developed. Even though the piezometers will not be sampled, they will be developed so they may be used for water level measurements. The wells will be developed by a licensed drilling contractor using bailing, surging, and/or pumping techniques until the effluent water is relatively free of fine sediment and the total depth of the wells have been restored as close as possible to the depth recorded the last time the wells were sampled in 1999. Data to be collected during well development will include: initial and final well depth, initial and final depth to water, pumping rates, gallons purged, and groundwater indicator parameter readings including pH, electrical conductivity, temperature and turbidity.

The drilling contractor will also attempt to retrieve the stuck pump from well MW-2. If the pump retrieval is unsuccessful, then piezometer PZ-1 will be sampled in place of MW-2 since it is of similar depth and they are proximal to each other.

2.4.2 Instrument Calibration

Meters used to measure groundwater indicator parameters (i.e., conductivity, pH, temperature, dissolved oxygen, oxidation-reduction potential (ORP), and turbidity) will be rented from a reputable environmental equipment supplier. Immediately prior to shipping, the equipment will be pre-calibrated by the supplier according to manufacturers' recommendations. Field technicians will confirm that calibration documentation has been provided. The meters will be checked daily in the field to ensure the instruments remain within the proper calibration range.

2.4.3 Water Level Measurements

Before the existing monitoring wells and piezometers are purged, the depth to water will be measured from a surveyed point on the well casing using an electronic well sounder graduated to 100th of a foot. A detailed procedure for water level measuring is presented in WSP SOP#3 (Attachment A). All of the wells and piezometers will be surveyed by a licensed land surveyor relative to North American Vertical Datum 1988 (NAVD88).

2.4.4 Low-Flow/Minimal-Drawdown Purging Procedures

Each monitoring well will be purged until a representative sample of the groundwater from the water-bearing zone (WBZ) can be collected. Purge rates should be as slow as practical; ideally no faster than the rate that water re-enters the casing. The wells will be purged using a method that creates the least possible disturbance of the water in the well, given reasonable time constraints and the initial groundwater head conditions in the water.

The low flow/minimal drawdown purging method shall be performed using a clean submersible pump (e.g., electric submersible or air-bladder pump) with new polyethylene discharge tubing. The set up should include a controller capable of controlling the discharge rate of the pump to as low as 100 milliliters per min (mL/min). Typical purge rates for low-flow purging are between 200 and 500 mL/min, which are substantially below the rates used to develop wells. Low-flow purging should be performed with the use of a flow-through cell equipped with indicator parameter meters for measuring pH, conductivity, temperature, ORP, and dissolved oxygen. Turbidity is measured separately. If the well drawdown is at a continuous flow rate of 100 mL/min, the low-flow purging method will be abandoned and a standard purge will be performed (i.e. purge three casing volumes).

Low flow/minimal drawdown purging procedures are outlined in WSP SOP #3 (Attachment A) and will include the following:

- The pump should be lowered very slowly through the water column (<2 feet per minute) in order to cause the least possible disturbance of the water.
- The pump intake will be positioned approximately 5 feet from the well bottom to ensure the pump intake is within the well screen interval.
- Allow the well 15 minutes to stabilize before starting to purge.
- As a guideline, the drawdown in the lower WBZ wells should be limited to within 25% of the distance between the top of the well screen and the pump intake. For example, if the depth to the well screen is 50 feet and the depth of the pump intake is at 60 feet, then the well should not be drawn below a depth of 52.5 feet. This example assumes that the depth to water is 50 feet or less. If practical, meaning if slowing the purge rate down too much does not require an excessive amount of time to sample the well, the drawdown should be kept less than 0.3 feet to limit the stress on the well and allow for a sample to be collected with as little turbidity as possible. Water levels should be recorded regularly during purging to assure that the drawdown has stabilized.
- Each well will be purged until all indicator parameters have stabilized for three consecutive parameter readings and after at least four total parameter measurements have been recorded. Indicator parameters will be read at the start of purging and after each system volume has been removed. Purge volumes, times, water level measurements and indicator parameter measurements shall be recorded. Note: A system volume equals the volume of the discharge tubing between the pump and the flow-through cell plus the volume of the flow-through cell itself. The time interval between indicator parameter readings is calculated by dividing the purge rate into one system volume. Stability will have been reached when three consecutive readings are consistent with the following ranges: pH (+/- 0.1 pH units), EC (+/- 3%), temperature (+/- 3%), ORP (+/- 10 mV) and dissolved oxygen (+/-10%). If possible, the purge will continue until turbidity is less than 10 NTU at the time of sampling. All the indicator parameter data and system volume calculations will be record on the Groundwater Purging and Sampling Log (Attachment C).
- After the indicator parameters have stabilized, the flow through cell will be disconnected from the system and the sample bottles will be filled from the water stream coming out of the discharge tubing. The flow rate of the discharge while sampling will be no faster than the rate at which the well was purged and will be a maximum of 500 mL/min.
- Purge water will be pumped directly into a temporary storage tank and offloaded at the end of the sampling and purging event to Quemetco's wastewater treatment plant, where the purge water will be treated.

2.4.5 Sample Collection

Sampling shall be done in a systematic, consistent manner. Samples shall be collected in clean, pre-preserved laboratory-supplied containers as specified in SOP #2 (Attachment A) and indicated on Table A2.

Containers shall be clearly labeled as follows:

- Site name
- Well or identification number
- Preservative type and amount
- Date and analysis required for the sample.

Samples shall be immediately placed in a pre-chilled cooler to be shipped and analyzed promptly by the specified laboratory. All samples and labeling data shall be recorded on the field sampling form with additional information included in the field notebook, as necessary.

2.4.6 Groundwater Analytical Program

Groundwater samples will be analyzed by Pace Analytical for the following parameters (Table A1):

- Total metals (aluminum, antimony, arsenic, barium, cadmium, calcium, chromium, copper, lead, magnesium, mercury, nickel, sodium, zinc) by EPA methods 6020/7470.
- Dissolved metals (aluminum, antimony, arsenic, barium, cadmium, calcium, chromium, copper, lead, magnesium, mercury, nickel, sodium, zinc) by EPA methods 6020/7471. Note: Samples collected for dissolved metals analysis will be filtered in the field using a 0.45 micron, in-line, filter before decanting into laboratory supplied containers.
- Total phenols by EPA Method 8270C
- Total suspended solids by EPA Method SM 2540 D

The analytical report will include a Level IV data package.

2.4.7 Quality Assurance/Quality Control

For QA/QC purposes, duplicates and MS/MSD samples will be collected and analyzed at a minimum frequency of one duplicate per 20 samples and one MS/MSD per 20 samples. Duplicate samples shall be taken in the same manner as the initial samples and identified as a separate well or with a unique identification that does not indicate the initial sample identification.

The submersible pump will be decontaminated before initial use and after each sampling location in accordance with SOP #16. One equipment blank sample will be collected from the submersible pump in a manner that duplicates well sampling as much as practical. The location and the ambient conditions at the location where the equipment blank was collected will be documented in the field notes.

A summary of QA/QC sample collection and analysis is provided in the QAPP (Appendix B of the Workplan). The field QA/QC samples will be collected in accordance with SOP #21.

3 DERA Investigation

This section presents the DERA field sampling plan and analytical methods. Soil, sediment, surface water, and benthic invertebrate samples will be collected from various locations throughout the Site to be used for chemical and the ecological-biological-toxicology analysis. The chemical of concern is predominately lead; however, this project will also address the possible impacts of other chemicals of concern. A summary of the DERA sampling activities is included on Table A1.

3.1 Decision Statement

The DERA field sampling will include benthic invertebrates and toxicity tests using soil, sediments, and surface water. The proposed sampling locations for the DERA are shown in Figure A5.

Results from the chemical analyses for sediment, soil, and surface water will be used to supplement the ecological-biological-toxicology results. Chemical results for soils will not be compared to ecological screening levels for soils (EcoSSLs). Chemical results will be used to evaluate the statistically significant affects and any potential correlations between ecological-biological-toxicology results from the Ponds and upstream and downstream of Julia Creek. Target parameters (laboratory reporting limits), will be reviewed and all applicable data will be incorporated into the revised DERA, as necessary.

The DERA addendum will be completed to incorporate all new data collected and will assess new receptors (i.e., Indiana bat, sandpiper) and reassess the risks to the American robin by incorporating new data. These results will be incorporated into the weight-of-evidence evaluation in the Risk Characterization component of the DERA addendum and will also include updating exposure parameters and toxicity reference values, as needed.

DERA's generally use the lowest-observed-adverse-effect levels (LOAELs) for determining the need for remediation. Therefore, LOAELs have been used to derive Ecological Action Levels for the Quemetco site (Intrinsik memo to EPA, February 2014). The Ecological Action Levels for the Quemetco site were derived by using the toxicity data for wildlife compiled within EPA's Eco-SSL document (EPA, 2005). The agreed upon Ecological Action Level for lead for the Quemetco site (130 mg/kg) will be used for screening and will be further assessed in the addendum to the DERA.

3.2 Collection of Soil Samples

As indicated in Section 3.2, pages 10-11 of the Workplan, soil samples will be collected for chemical analysis from each of the following locations (Figure A5):

- P1-13 through P1-16 near Pond No. 1
- P2-10 through P2-13 near Pond No. 2
- P3-16 through P3-20 near Pond No. 3
- JCS-1 through JCS-9 and JCS-18 through JCS-23 along Julia Creek

Soil samples will be collected using hand trowels, spoons or shovels in accordance with SOP#11 (Attachment A). Information on the number and size of soil sample containers and the storage

requirements are included on Table A2 and in SOP #2. All soil samples will be collected using decontaminated stainless steel equipment. All vegetation will be removed prior to sample collection at each location. Soil samples will be collected from 0 to 6 inches bgs. When sampling at a location has been completed, all equipment will be decontaminated according to the procedures set forth in Section 7 of this SAP and SOP #19.

The following materials will be used when collecting soils samples:

- Field log book
- Personal Protective Equipment (PPE)
- Stainless steel trowels or spoons
- Shovel
- Mixing tray or bowl
- Sample Containers

The following procedures will be adhered to during soil sample collection with a stainless steel trowel or shovel:

1. Use appropriate PPE as specified in the site-specific HASP.
2. Remove any vegetation or other surface material (e.g., gravel) from the sample location with a trowel or other tool (e.g., shovel).
3. Push the trowel into the soil to the desired sampling depth. If sampling a stiff silty or clayey soil, it may be necessary to remove and reinsert the trowel to loosen the soil. Shallow subsurface soil samples can be collected by digging a hole (e.g., with a shovel or trowel) and collecting a soil sample at the desired depth.
4. Transfer the soil to a mixing tray or bowl using a decontaminated stainless steel trowel or spoon.
5. Describe the soil in accordance with ASTM International Standard D 2488 and the Unified Soil Classification System. Record the soil description in the field logbook.
6. Use the stainless steel spoon or trowel to chop apart clumps of soil material and mix the contents of the tray to a homogeneous particle size and texture.
7. Examine contents of the tray and remove pebbles, organic material, (e.g., roots, grass, and woody material), and other debris with a stainless steel trowel or spoon.
8. Transfer the tray contents to the appropriate sample container using a stainless steel spoon.
9. Label the containers, cover the labels with tape, and immediately place the containers in a cooler maintained at an ambient temperature of 4 degrees Celsius with wet ice. Freezer packs or dry ice should not be used for sample preservation.
10. Record the sample location, sample depth, sampler name, and the requested analytical parameters in the field log book.
11. Complete the chain-of-custody form with appropriate sampling information.
12. Samples should be maintained and shipped in accordance with the procedures described in SOP #20

13. All PPE and IDW shall be managed in accordance with state and federal requirements.

3.2.1 Analytical Program

Soil samples collected for laboratory analyses of conventional chemical parameters will be sent to Pace Analytical and will include the following parameters (Table A1):

- Total metals (antimony, arsenic, barium, cadmium, chromium, copper, lead, mercury, nickel, zinc) by EPA Method 6020/7471
- pH by EPA Method 9045
- Total Organic Carbon by Walkley-Black method (EPA 9060A)
- Soil moisture content by Method SM 2540 G
- Water holding capacity by Method SM 2540 G
- Cation exchange capacity by EPA Method 9081

3.2.2 Field QA/QC Samples

For quality control and quality assurance purposes, duplicates and MS/MSD samples will be collected and analyzed at a minimum frequency of one duplicate per 20 samples and one MS/MSD per 20 samples as referenced in SOP#21. Additionally, equipment blank samples will be collected from the stainless steel trowel/spoon and mixing trays. A summary of QA/QC sample collection and analysis is provided in the QAPP (Appendix B of the Workplan).

3.3 Ecological-Biological-Toxicology Assessment

Soil samples will be collected from several areas of the Site to evaluate the potential for effects of metals on earthworms in these areas. Earthworms and other soil invertebrates serve many ecologically important functions, including aeration of the soil, litter breakdown, and they are food for mammalian and avian wildlife. To evaluate the toxicity of metals to earthworms, soil will be collected around Pond Nos. 1, 2, and 3, Julia Creek, the Slag Pile, the NE Area and previous background sampling areas. Composite soil samples will be collected from 0 to 6 inches bgs.

The locations of the soil samples collected for earthworm toxicity testing are presented on Figure A5.

3.3.1 Collection of Composite Soil Samples

Composite soil sample collection procedures will consist of the collection of double volume grab soil samples from 0 to 6 inches bgs as described in SOP#11 and SOP#32. The soil will be homogenized and the required amount will be placed in the sample containers for chemical analysis for the indicated sampling locations described in the Workplan Section 3.5, pages 12-13. The remaining soil will be placed in a second bag to be homogenized with soil samples collected from the remaining soil sample locations in this area to form the composite soil sample.

For example, composite sample COMP-1 near Pond No. 1, will consist of a soil sample from location P1-13 and continue to P1-16. The soil will be collected from 0 to 6 inch bgs, homogenized, and placed in a plastic bag to be homogenized with soil collected from 0 to 6 inches bgs from sample locations P1-14, P1-15 and P1-16. Enough subsurface soil will collected in total to fill all required sample containers for COMP-1. These procedures will be conducted for each successive composite sample collected as follows (Figure A5):

- COMP-2 near Pond No. 2 beginning with P2-10 and continuing with P2-11, P2-12 and P2-13.
- COMP-3 near Pond No. 3 beginning with P3-16 and continuing with P3-17, P3-18, P3-19 and P3-20
- COMP-JC along Julia Creek beginning with JCS-1 and continuing with JCS-3, JCS-4, JCS-5, JCS-18, JCS-20, JCS-22 and JCS-23
- COMP-SP from the Slag Pile beginning with COMP-SP1 and continuing with COMP-SP2, COMP-SP3, and COMP-SP4.
- COMP-NE near the northeast area of the Site beginning with COMP-NE1 and continuing with COMP-NE2, COMP-NE3 and COMP-NE4.
- COMP-BK from the southwest area of the Site beginning with BSB-1 and continuing with BSB-9 and BSB-10.

Detailed soil composite sampling procedures are presented in SOP #32 and the specific sample containers, preservatives and handling procedures are presented in Table A2 and SOP #2. All composite soil sampling locations are included on Figure A5.

3.3.2 Analytical Program

Composite soil samples will be used in the evaluation of earthworm toxicity by U.S. EPA protocol 600/3-88-029, a 14-day toxicity test that evaluates survival and biomass of earthworms (Table A1). Earthworm toxicity testing will be conducted by NAS. Additionally, the composite soil samples will be analyzed for particle size analysis by Method ASTM D 422-Atterberg Limits by Pace Analytical.

3.3.3 Field QA/QC Samples

Equipment blank samples will be collected from the stainless steel trowel/spoon and mixing trays as specified in SOP #21. A summary of QA/QC sample collection and analysis is provided in the QAPP (Appendix B of the Workplan).

3.4 Sediment Investigation

An investigation will be conducted to characterize current sediment conditions, including potential chemical and toxicity levels in Julia Creek and Pond Nos. 1, 2, and 3 (Figures A2 and A5). Results from the sediment sampling will be used in the Effects Assessment component of the DERA.

3.4.1 Collection of Sediment Samples

The sediment sampling event will be conducted concurrent with the soil sampling activities. Sediment samples will be collected for chemical and toxicity testing from the following locations:

- SD-9 and SD-10 near Pond No. 1
- SD-11, SD-12 and SD-13 near Pond No. 2
- SD-14 through SD-17 near Pond No. 3
- SD-1 through SD-8 and SD-18 through SD-23 for a total of 14 samples along Julia Creek

At all locations, sediment samples will be collected from the sediment surface at approximately 0 to 0.5 feet of the sediment layer. Depending on the depth of water at the time of sampling, the shallow sediment samples will be collected using a stainless steel scoop, Ponar or Eckman dredge, or hand corer in accordance with SOPs # 4, 5 and 6. Sample equipment refusal will be indicative of the presence of the underlying native soil. Additionally, all sample equipment will be decontaminated before initial use and between sampling locations.

Interstitial pore water ammonia and interstitial pore water pH will be analyzed in sediment samples. Interstitial pore water is defined as the water occupying spaces between sediment particles. Interstitial pore water may occupy 50% or greater of the volume of a sediment sample and is in contact with sediment surfaces for long periods of time. Therefore, interstitial pore water analytical results may be indicative of the condition of ground or surface waters at the Site. Interstitial pore water samples will be collected ex-situ by using the core method, Ekman or Ponar dredge or hand trowel (SOP#6, SOP#5, and SOP#4, respectively) and shipped to RTI Laboratories in Livonia, Michigan for ex situ pore water extraction via centrifugation. The common laboratory procedure for collecting interstitial pore water samples in sediment is presented in SOP #33. Specific information on the number and size of sample containers and any preservatives or handling requirements are included in SOP #2.

Sediment samples will be collected after all surface water samples are collected. Sediment sampling will begin with the most downstream location and will progress upstream to minimize the possibility of cross-contamination.

Depending on the depth of water at the time of sampling, field parameters will be collected for sediments in accordance with SOP 4, 5 and 6. Field parameters collected during the sediment sampling activities will include the following:

- pH
- Visual observations including color, texture, presence of oil sheens, macrophites or animals

3.4.2 Analytical Program

Sediment samples SD-1 through SD-23 will be collected in specific sample containers, preservatives and handling procedures according to Table A2 and SOP #2. Sediment samples will be analyzed by Pace Analytical for the following parameters (Table A1):

- Total metals (antimony, arsenic, barium, cadmium, chromium, copper, lead, mercury, nickel, zinc) by EPA Method 6020/7471

- Total Phenols by EPA Method 8270C
- AVS/SEM by EPA Method 1629/6010
- TOC by Walkley-Black method
- Particle size analysis by ASTM D 422-63;
- Interstitial pore water ammonia by EPA Method 350.1
- Interstitial pore water pH by EPA Method SM 4500H-B
- cation exchange capacity by EPA Method 9081

Sediment samples will be collected for analysis by NAS for the amphipod crustacean *Hyalella azteca* (*H. azteca)* (28-day toxicity) by EPA/600/R-99/064 100.1/100.4 and C. *dilutus* (10-day) toxicity tests by EPA/600/R-99/064 -100.1/100.2.

3.4.3 Field QA/QC Samples

For QA/QC purposes, duplicate and MS/MSD samples will be collected and analyzed at a minimum frequency of one duplicate per 20 samples and one MS/MSD per 20 samples. A summary of QA/QC sample collection and analysis is provided in the QAPP (Appendix B of the Workplan). Additionally, one equipment blank sample will be collected from the stainless steel scoop, Ponar or Eckman dredge, or hand corer used to collect sediment samples, as applicable and in accordance with SOP #21.

3.5 Surface Water Investigation

Based on the results of sampling and analysis from 2006, two additional surface water samples from Pond No. 3 and six additional surface water samples from Julia Creek will be collected for chemical analysis and toxicity testing (Figure A5).

The surface water samples collected from Pond No. 3 and Julia Creek will be transported by overnight mail service or courier to Pace Analytical for chemical analysis. The surface water samples will be sent to NAS for toxicity testing. Details on the specific analyses for each sample are listed below and presented in Table A1.

3.5.1 Collection of Surface Water Samples

The surface water sampling events will be conducted concurrent with the soil and sediment sampling activities. Surface water sampling will begin with the most downstream location and will progress upstream to minimize the possibility of cross-contamination. The samples will be collected from the bank of the ditch, if possible. Caution will be taken to limit turbulence in the sample areas. Samples will generally be collected directly into the appropriate sample containers. The container will be placed in the flowing water just beneath the stream surface. In the event that the sample container has been pre-preserved at the laboratory, a new clean glass jar will be used to transfer the sample from the flowing water to the pre-preserved sample container. Surface water samples will be collected in accordance with the QAPP (Appendix B of the Workplan) and SOP #4.

The surface water samples include the following (Table A2 and Figure A5):

- P3-SW-1 and P3-SW-2 from Pond No. 3. One surface water sample will be collected from the south side of Pond No. 3 (P3-SW-1) and one from the north side of Pond No. 3 (P3-SW-2).
- SW-1 through SW-8, SW-18 through SW-23 from Julia Creek will be submitted for chemical, pH and water hardness analysis.
- SW-1, SW-3, SW-4, SW-5, SW-18, SW-20, SW-22 and SW-23 from Julia Creek will be submitted for toxicity testing using P. *promelas* and C. *dubia.*

In addition to the laboratory analyses that will be performed (Table A1), the following field parameters will be measured in situ at each sampling location:

- pH
- temperature
- specific conductance
- turbidity
- dissolved oxygen (DO)
- redox potential

The field testing will be performed to evaluate those parameters (excluding specific conductance and turbidity) that will change ex-situ and for comparison with nearby groundwater conditions. Because these data are to be used for qualitative purposes, only pH will have confirmatory laboratory analysis performed.

3.5.2 Analytical Program

Surface water samples P3-SW-1, P3-SW-2, SW-1 through SW-8, and SW18 through SW-23 will be collected in specific sample containers, preservatives and handling procedures according to Table A2 and SOP #2. Surface water samples will be analyzed by Pace Analytical for the following parameters:

- pH by SM4500 H;
- Water hardness by SM 2340B
- Total metals (aluminum, antimony, arsenic, barium, cadmium, calcium, chromium, copper, lead, magnesium, mercury, nickel, sodium, zinc) by EPA Methods 6020/7470
- Dissolved metals (aluminum, antimony, arsenic, barium, cadmium, calcium, chromium, copper, lead, magnesium, mercury, nickel, sodium, zinc) by EPA Methods 6020/7470

Surface water samples collected from P3-SW-1, P3-SW-2, SW-1, SW-3, SW-4, SW-5, SW-18, SW-20, SW-22 and SW-23 will be analyzed by NAS for the following parameters:

- Fathead minnow (*P. promelas*) and water flea *(C. dubia*) (chronic) toxicity test by EPA-821-R-02-013 Method 1000.0

3.5.3 Field QA/QC Samples

For the chemical analyses, duplicate and MS/MSD samples will be collected and analyzed at a minimum frequency of one duplicate per 20 samples and one MS/MSD per 20 samples. No QA/QC samples will

be collected for the toxicity testing. A summary of QA/QC sample collection and analysis is provided in the QAPP (Appendix B of the Workplan). The field QA/QC samples will be collected in accordance with SOP #21.

3.6 Benthic Invertebrate Sampling

Benthic invertebrates will be sampled in Julia Creek and Pond Nos. 1, 2, and 3 for tissue analysis and the results will be used in the risk characterization in the DERA. Benthic invertebrates will be collected from each of the 16 sediment sampling locations (Figure A5). WSP will retain ASC Group, Inc. from Indianapolis, Indiana, a firm familiar with invertebrate sampling in Marion County and WSP will assist in the collection of the samples.

3.6.1 Collection of Benthic Invertebrate Samples

Sampling for benthic invertebrates will be conducted in accordance with SOP #34. It is anticipated that the sampling for the invertebrates will stir up bottom sediments significantly and so they will not be collected until after the surface water and sediment samples have been collected. Also, the sampling will be conducted in a downstream to upstream direction in Julia Creek.

Benthic invertebrate sampling will be conducted using d-frame kick nets. Sampling procedures are discussed in detail in SOP #34 and will include the following:

- In the riffle kick portion of the stream, locate an area in the lower (downstream) area of the riffle or run habitat that is to be sampled.
- While holding the d- frame net handle, the flat end of the d-frame net is placed securely onto the substrate so that there are no gaps under the frame.
- Disturb (by kicking) the riffle/run substrate one foot directly in front of the d-frame net so that any dislodged macroinvertebrates are carried by the current into the net.
- Move in an upstream or diagonal upstream direction across the riffle/run, continuously disturbing the substrate in front of the d- frame net, for a distance of about 10 feet (this is roughly equivalent to sampling one square meter of riffle/run habitat).
- After completion of the kick, inspect the contents of the net. Any stones (gravel sized or smaller) or debris (leaves, small sticks) that are kicked into the net are retained. Cobble and large sticks kicked into the net should be visually inspected and any attached macroinvertebrates removed and placed in the net; the cobble or large sticks are then returned to the stream.

3.6.2 Analytical Program

The 16 benthic invertebrate sampling locations will be identified as BI-1 through BI-5, BI-8, BI-9, BI-12, BI-13, BI-15, BI-17 through BI-20, BI-22, and BI-23. The benthic invertebrate samples will be collected in specific sample containers with preservative and handling procedures conducted according to Table A2 and SOP #2. Benthic invertebrate samples will be analyzed by Pace Analytical for the following parameters:

- Total metals (aluminum, antimony, arsenic, barium, cadmium, calcium, chromium, copper, lead, magnesium, mercury, nickel, sodium, zinc) by EPA Methods 6020/7470. Results will be reported in both wet weight and dry weight.

- Moisture content by ASTM D2974-87

3.6.3 Field QA/QC Samples

Duplicate benthic invertebrate samples will be collected for the chemical analyses at a rate of 1 per 10 if enough organisms are available. A trip blank will be included in each cooler. A summary of QA/QC sample collection and analysis is provided in the QAPP (Appendix B of the Workplan).

4 Sample Handling, Field Documentation, Equipment, Decontamination, and Waste Handling

This section describes the general procedures for field sample collection during the activities described in Section 1 through 3, how the samples will be prepared for delivery to the laboratories, recordation of field activities and data, how equipment will be decontaminated, and how investigation-derived waste will be handled. This section complements WSP SOPs presented in Attachment A.

4.1 Sampling Handling

To identify and manage samples obtained in the field, a sample label will be affixed to each sample container. The sample labels will include the following information:

- Site name
- Zone number (for XRF sampling)
- Sample identification number
- Sampler's initials
- Date and time of collection
- Preservative, if any

Following collection and labeling, samples will be immediately placed in a sample cooler for temporary storage. The following protocol will be followed for sample packaging:

1. Samples to be shipped will be placed in the cooler and packed with packaging materials to minimize the potential for disturbance and/or breakage of the sample containers.
2. Ice will be placed in leak-resistant plastic bags and added to the coolers to chill the samples during transportation to the analytical laboratory.
3. The chain-of-custody form will be placed in a water-resistant plastic bag and taped on the inside of the lid of the cooler.

All samples will be maintained in the custody of the sampling team. The samples will be transported to the analytical laboratory at the end of the sampling day under appropriate chain-of-custody procedures.

4.2 Field Documentation

4.2.1 Field Logbooks

Field logbooks will document where, when, how, and from whom any vital project information was obtained. Logbook entries will be bound with consecutively numbered pages. Each page will be dated and the time of entry noted in military time.

At a minimum, the following information will be recorded during the collection of each sample:

- Zone number (for XRF sampling)
- Sample identification number
- GPS location coordinates
- Date and time of sample collection
- Type of sample (i.e., matrix)
- Type of sampling equipment used
- Field observations and details important to analysis or integrity of samples (e.g., heavy rains, odors, colors, etc.)

4.2.2 Field Forms

Field forms will be used as an efficient and consistent way to record data and document field methodologies when developing and sampling groundwater monitoring wells (Attachment C).

4.2.3 Chain-of-Custody Records

Chain-of-custody (COC) records are used to document sample collection and shipment to the laboratory for analysis. All sample shipments will be accompanied by a COC record. The COC record will identify the contents of each shipment and maintain the custodial integrity of the samples.

4.3 Decontamination Procedures

All non-disposable sampling devices that are used during the sample collection process (pumps, troughs, shovels, collection trays, measuring tapes, etc.) for all media (soil, sediment, surface water, and groundwater) will be cleaned before initial use and between sample points. When such equipment is not in use it will be stored on plastic bags or other means to keep it clean. All non-disposable sampling devices will be decontaminated using the following procedures found in SOP #s 16, 17 and 19 (Attachment A):

- Non-phosphate detergent and tap water wash, using a brush if necessary to remove visible dirt
- Tap-water rinse
- Distilled water rinse

Disposal of dedicated equipment intended for one time use (polyethylene disposable bailers, discharge tubing, etc.) where applicable, will not be decontaminated. This equipment will be packaged in Quemetco's PPE refuse containers for appropriate disposal.

4.4 Investigation Derived Waste Management

In the process of collecting environmental samples during the proposed field sampling program, different types of potentially contaminated investigation-derived wastes (IDW) will be generated. The wastes will be handled as follows:

- Soil will be placed back into their respective sample holes.

- Decontamination fluids will be collected and processed through the wastewater treatment plant.
- Groundwater monitoring well development and purge water will be collected in 55-gallon steel drums and properly stored onsite, pending waste profile. Upon receipt of groundwater monitoring data, and if the collected groundwater is determined to be non-hazardous, the development and purge water will be treated in Quemetco's onsite wastewater treatment plant. The IDW will be managed in accordance with WSP SOP #26.
- Used PPE and disposable equipment (sampling trays, towels, polyethylene bailers and string) will be placed in Quemetco, Inc.'s PPE refuse containers. Used PPE will be handled as hazardous waste consistent with Quemetco, Inc.'s waste handling practices.

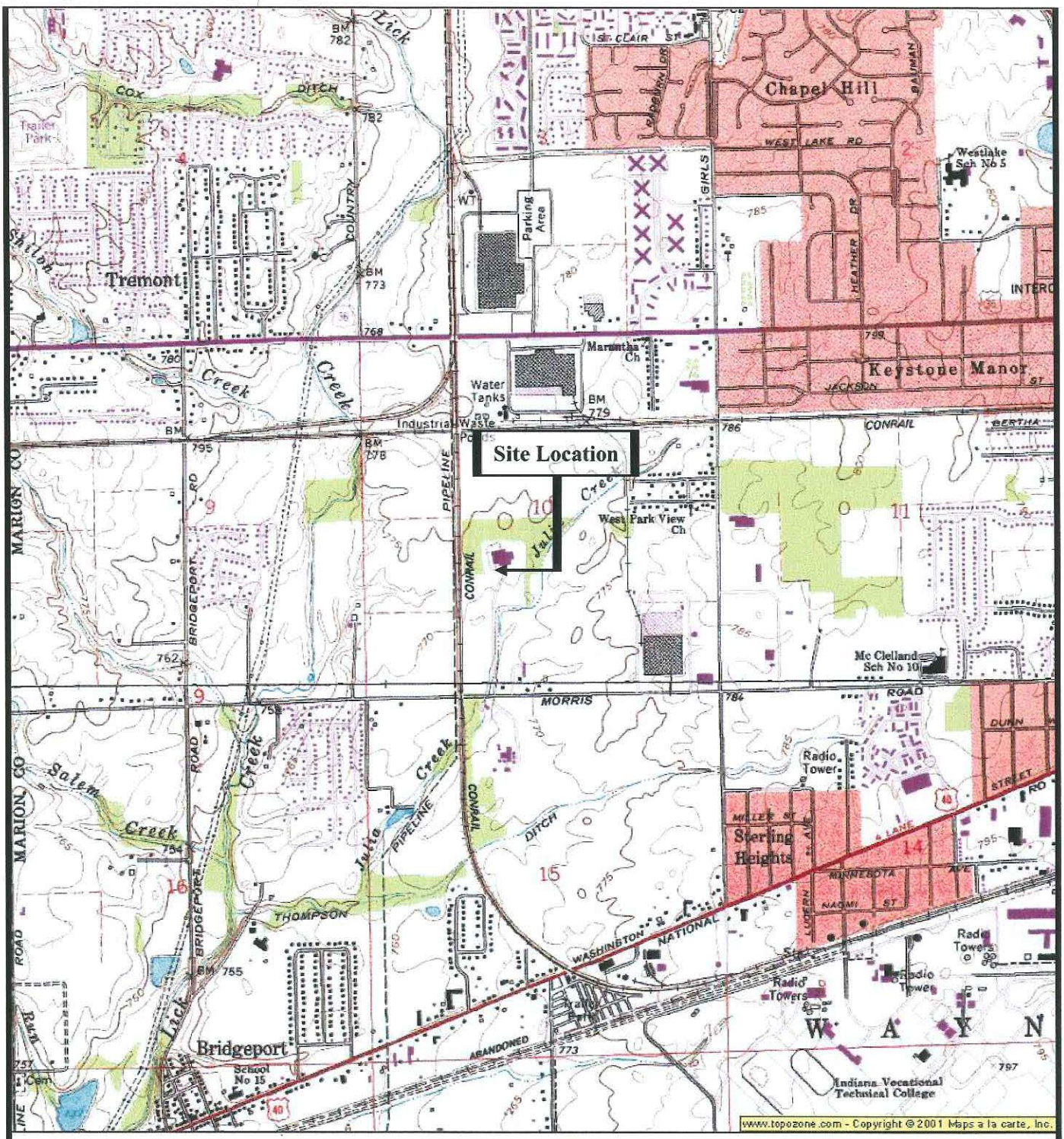
5 Reporting

Upon completion of the sampling activities and receipt of laboratory data, a report detailing the investigation efforts will be prepared and submitted to U.S. EPA. The report will include sample methodology, sample results, laboratory analytical and QA/QC review, and a map depicting GPS-recorded sample locations.

Amy Romano, senior technical manager of WSP will be the WSP Quality Assurance Officer (QAO) during the project and will oversee all QA/QC aspects of the work. Terrie Baranek of ECT.CON, Inc. will be the third-party data validator for the fixed laboratory chemical analyses. Ms. Ruth Hull of Intrinsik will review the data generated from the ecological-biological-toxicity testing. The WSP QAO will remain independent of direct job involvement and day-to-day operations, report to the WSP Project Director, and will resolve any QA disputes. The third-party data validator will report to the WSP Project Manager.

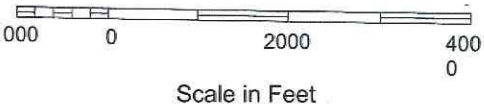
Figures

q:\client\quemetco\indiana\indianapolis, in figure 1



Reference
7.5 Minute Series Topographic Quadrangle
Clermont, Indiana
Photorevised 1984 Scale 1:25,000

Quadrangle Location



Scale in Feet

WSP
2025 Gateway Place, Suite 435
San Jose, California
(408) 453-6100

Figure A1
Site Location
Location of Quemetco Facility
Indianapolis, Indiana

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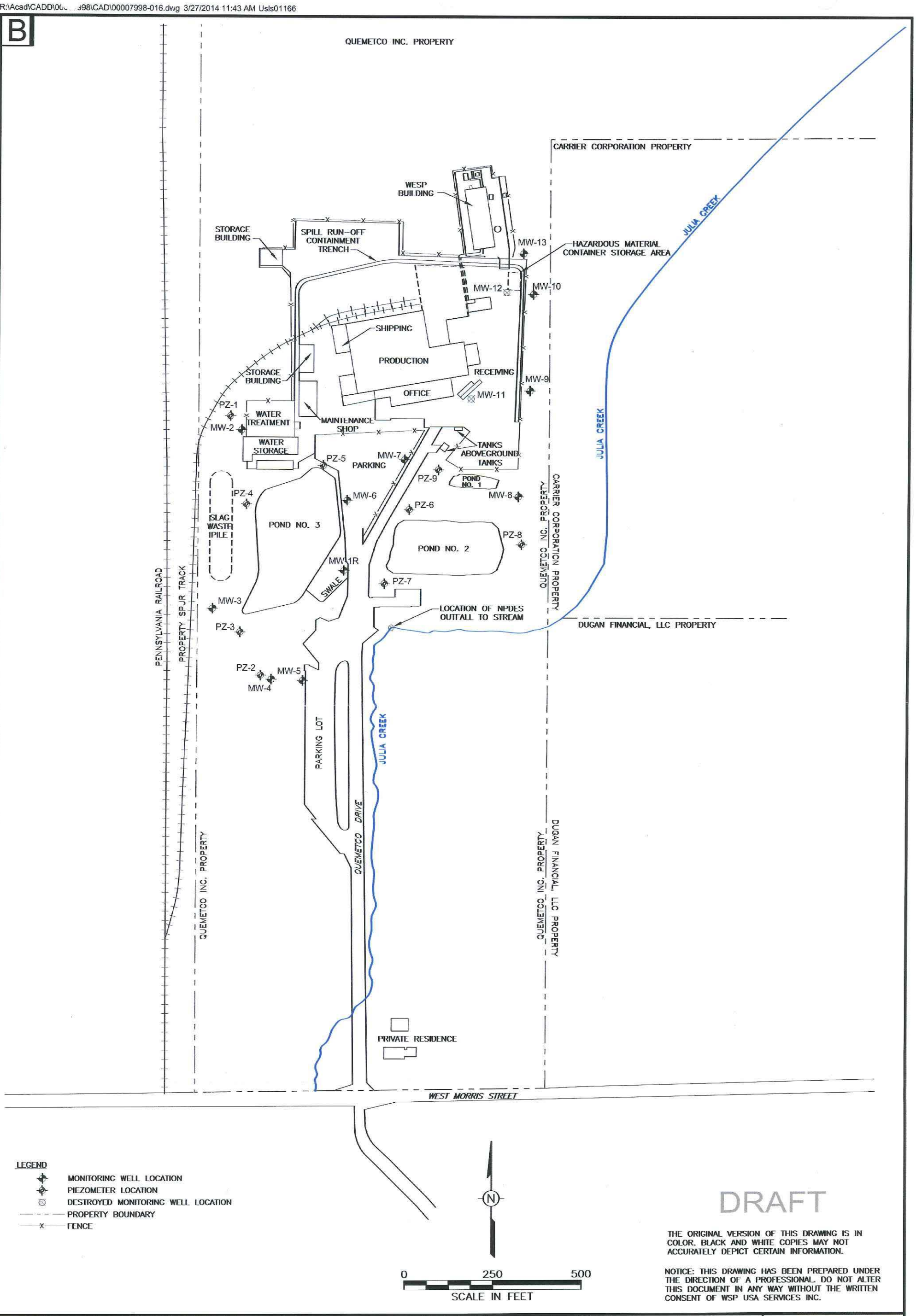

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www.wspgroup.com/usa

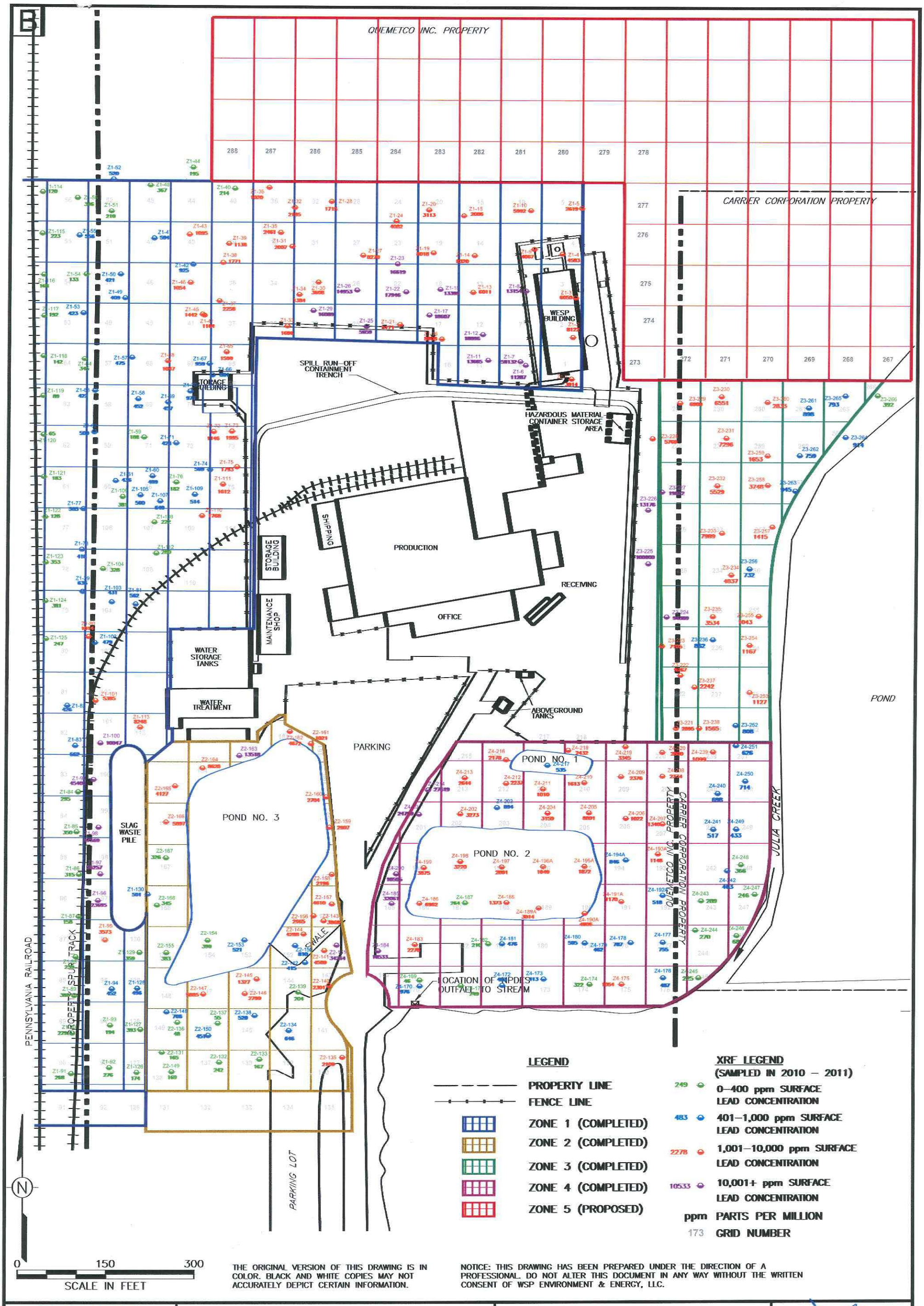
Figure A2

SITE PLAN

900 QUEMETCO DRIVE
INDIANAPOLIS, INDIANA

PREPARED FOR
QUEMETCO, INC.
INDIANAPOLIS, INDIANA

Drawn By:	LS 3/26/2014
Checked:	
Approved:	
DWG Name:	00007998-016



THE ORIGINAL VERSION OF THIS DRAWING IS IN COLOR. BLACK AND WHITE COPIES MAY NOT ACCURATELY DEPICT CERTAIN INFORMATION.

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Figure A3

PROPOSED XRF SAMPLING GRID

900 QUEMETCO DRIVE
INDIANAPOLIS, INDIANA

PREPARED FOR
QUEMETCO, INC.
INDIANAPOLIS, INDIANA

Drawn By:	DJC	7/06/2012
Checked:	AW	7/13/2012
Approved:	BJM	7/13/2012
DWG Name:	00007998-014	

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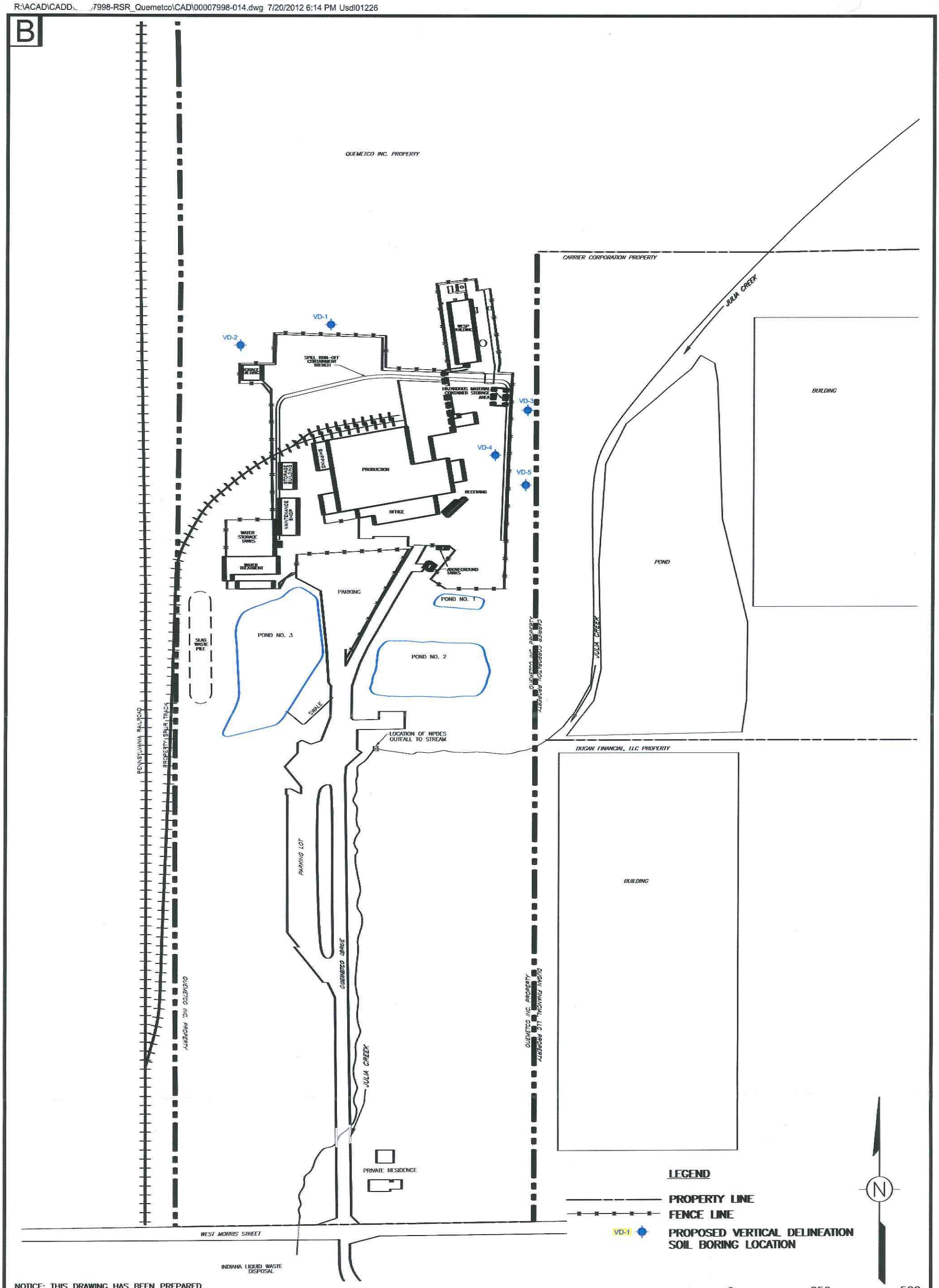

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Figure A4

PROPOSED SOIL BORINGS FOR VERTICAL DELINEATION OF LEAD

900 QUEMETCO DRIVE
INDIANAPOLIS, INDIANA

PREPARED FOR
QUEMETCO, INC.
INDIANAPOLIS, INDIANA

Drawn By:	DJC	7/06/2012
Checked:	AW	7/13/2012
Approved:	BJM	7/13/2012
DWG Name:	00007998-014	

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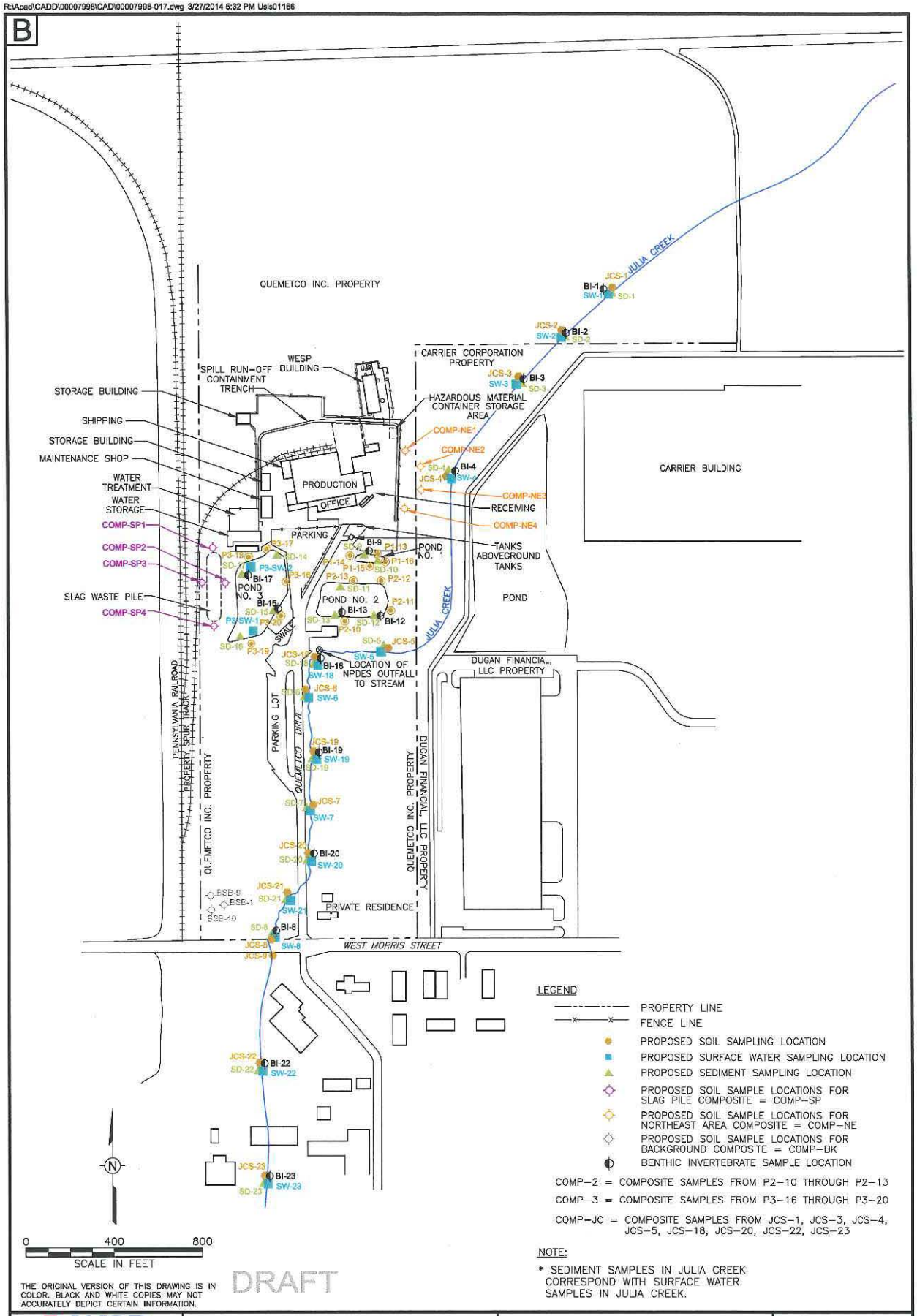

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Figure A5

PROPOSED DERA SAMPLING LOCATIONS

900 QUEMETCO DRIVE
INDIANAPOLIS, INDIANA

PREPARED FOR
QUEMETCO, INC.
INDIANAPOLIS, INDIANA

Drawn By: LS 3/26/2014
Checked:
Approved:
DWG Name: 00007998-017

Table A2

Table A2-Sample Container and Preservat[illegible] for Site Characterization and DERA Sampling

							Laboratory QA/QC control limits							
Sample Media	Analysis	Method	Holding Time	Container type/size	Preservative	Storage Requirement	Method Detection Limits (MDL)	Reporting Limits (RL/PQL)	LCS/LCSD Percent Recovery	LCS/LCSD Relative Percent Difference (RPD)	MS/MSD Percent Recovery	MS/MSD Relative Percent Difference (RPD)	Laboratory	Notes
surface soil	Metals						MDL (mg/kg)	PQL (mg/kg)						
	Antimony	EPA 6020	6 months	4 oz jar	none	cool, <6°C	0.015	0.10	80-120	20	75-125	20	PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9408	SEE ADDITIONAL NOTES AT BOTTOM OF TABLE
	Arsenic						0.013	0.10	80-120	20	75-125	20		
	Barium						0.009	0.10	80-120	20	75-125	20		
	Cadmium						0.011	0.10	80-120	20	75-125	20		
	Chromium						0.0166	0.10	80-120	20	75-125	20		
	Copper						0.047	0.10	80-120	20	75-125	20		
	Lead						0.008	0.10	80-120	20	75-125	20		
	Nickel						0.046	0.10	80-120	20	75-125	20		
	Zinc						0.856	2.00	80-120	20	75-125	20		
	Mercury	EPA 7471A/B	28 days				0.0032	0.007	85-115	20	85-115	20		
surface soil	pH	EPA 9045	ASAP	Three 4oz jars; 1 gallon zip lock bag	none	cool, <6°C	0.01 Std units	0.1 Std units	NA	5	NA	NA	PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9408	water holding capacity and Particle Size subcontracted to CQM, INC.; CEC, Pace -Kansas
	Total Organic Carbon (TOC)	EPA 9060A	28 days				73.37 mg/kg	600 mg/kg	80-120	30	50-150	20		
	Soil Moisture Content	SM 2540G	NA				0.10%	0.10%	NA	10	NA	NA		
	Water Holding Capacity		NA											
	Particle size analysis	ASTMD422/ Atterberg Limit					NA	NA		NA				
	Cation Exchange Capacity (CEC)	EPA 9081	180 days				0.05 meq/100g	0.1 meq/100g	62-138	47	NA	NA		
soil	14-day earthworm survival and biomass toxicity test	EPA 600/3-88-029	72 hr recommended (see footnote for reference)	plastic or PTFE containers; can use 1L or gal etc.; need 2 L	None. Fill container completely, i.e., no headspace. **Chill to 4°C. Do not freeze.**	Store at 4°C in the dark until used for testing.	N/A	N/A	N/A	N/A	N/A	N/A	Northwestern Aquatic Sciences 3814 Yaquina Bay Road Newport, Oregon 97365 Phone: 541.265.7225 Fax: 541.265.2799 contact@nwaquatic.com	testing 100% soil or 50% dilution series? Volume required depends on it. 1200g needed ~2L
XRF[1] soil	Lead	EPA 6020	6 months	4 oz jar	none	cool, <6°C	0.008 mg/kg	0.10 mg/kg	80-120	20	75-125	20	PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9406	soil samples collected every 10 XRF[1]
subsurface soil	Lead	EPA 6020	6 months	4 oz jar	none	cool, <6°C	0.008 mg/kg	0.10 mg/kg	80-120	20	75-125	20	PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9407	
surface water	Fathead minnow (P. promelas) and water flea (C. dubia) (chronic) toxicity test	EPA-821-R-02-013 Method 1000.0	48 hr or as soon as possible after collection	5 gal cubitainer of a grab sample	None. Fill container completely, i.e., no headspace. Chill to 4°C. Do not freeze.	Store at 4°C in the dark until used for testing.	N/A	N/A	N/A	N/A	N/A	N/A	Northwestern Aquatic Sciences 3814 Yaquina Bay Road Newport, Oregon 97365 Phone: 541.265.7225 Fax: 541.265.2799 contact@nwaquatic.com	testing 100% surface water or 50% dilution series? 5-gal will cover dilution series. Can only run 5 or 6 samples at one time if doing dilution series.
surface water	pH	SM 4500-H+	ASAP	2 plastic 250 ml	1- none; 1 HNO3 <2pH	cool, <6°C	0.01 Std Units	0.1 Std Units	NA	5	NA	NA	PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9407	
	water hardness	SM 2340B	6 months	2 plastic 250 ml	1- none; 1 HNO3 <2pH		150 ug/l	2000 ug/l	NA	20	NA	NA		
surface water	Metals (Total and Dissolved)						MDL (ug/L)	PQL (ug/L)						
	Antimony	EPA 6020	6 months	2 plastic 250 ml	pH<2; HNO3	cool, <6°C	0.115	1	80-120	20	75-125	20	PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9408	It is assumed that water samples will be field filtered for dissolved metals
	Arsenic						0.193	1	80-120	20	75-125	20		
	Barium						0.139	1	80-120	20	75-125	20		
	Cadmium						0.130	1	80-120	20	75-125	20		
	Chromium						0.1156	1	80-120	20	75-125	20		
	Copper						0.381	1	80-120	20	75-125	20		
	Lead						0.061	1	80-120	20	75-125	20		
	Nickel						0.155	1	80-120	20	75-125	20		
	Zinc						4.069	10	80-120	20	75-125	20		
	Mercury	EPA 7470A	28 days				0.100	0.200	85-115	20	85-115	20		
sediment	Metals						MDL (mg/kg)	PQL (mg/kg)						
	Antimony	EPA 6020	6 months	4 oz glass jar	none	cool, <6°C	0.015	0.10	80-120	20	75-125	20	PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9407	
	Arsenic						0.013	0.10	80-120	20	75-125	20		
	Barium						0.009	0.10	80-120	20	75-125	20		
	Cadmium						0.011	0.10	80-120	20	75-125	20		
	Chromium						0.0166	0.10	80-120	20	75-125	20		
	Copper						0.047	0.10	80-120	20	75-125	20		
	Lead						0.008	0.10	80-120	20	75-125	20		
	Nickel						0.046	0.10	80-120	20	75-125	20		
	Zinc						0.856	2.00	80-120	20	75-125	20		
	Mercury	EPA 7471A/B	28 days				0.0050	0.020	85-115	20	85-115	20		
sediment	Phenols Analyte						True MDL (µg/kg)	PQL (µg/kg)						
	2,3,4,6-Tetrachlorophenol	EPA 8270C					88.92	333	10-150	20		40	PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9407	
	2,4,5-Trichlorophenol						35.47	167	66-130	20		21		
	2,4,6-Trichlorophenol						42.46	167	68-130	20		33		
	2,4-Dichlorophenol						27.8	167	66-130	20		31		
	2,4-Dimethylphenol						31.93	167	66-130	20		29		
	2,4-Dinitrophenol						67.69	333	11-130	20		40		
	2-Chlorophenol						30.08	167	58-130	20		31		
	2-Methylphenol(o-Cresol)						32.59	167	65-130	20		29		
	2-Nitrophenol						32.59	167	63-130	20		41		
	3&4-Methylphenol(m&p Cresol)						24.38	167	63-130	20		29		
	4,6-Dinitro-2-methylphenol						17.52	167	33-130	20		50		
	4-Chloro-3-methylphenol						32.35	167	66-130	20		31		
	4-Nitrophenol						44.78	167	51-130	20		47		
	Cresols (Total)						56.97	333	10-150	20		40		
	Pentachlorophenol						22.54	167	43-130	20		50		
	Phenol						51.5	167	62-130	20		30		
sediment	*H. azteca* (28-day; survival and growth; and *C. dilutus* (10-day) toxicity tests	EPA/600/R-99/064 100.4 and 100.2	Normally 2 weeks; possibly up to 8 wks stored under nitrogen.	plastic or PTFE containers; can use 1L or gal etc.; need 5 L (see footnote for reference)	None. Fill container completely, i.e., no headspace. **Chill to 4C. Do not freeze.**	Store at 4°C in the dark until used for testing.	N/A	N/A	N/A	N/A	N/A	N/A	Northwestern Aquatic Sciences 3814 Yaquina Bay Road Newport, Oregon 97365 Phone: 541.265.7225 Fax: 541.265.2799 contact@nwaquatic.com	5L needed to do both a 10-d midge and 28-d Hyalella; allows for potential retest
sediment	Interstitial pore water Ammonia	EPA 350.1	28 days	Sediment -3 gallon covered bucket; Pore Water 250 ml plastic	H2SO4; <2pH	cool, 4°C	0.250 mg/L	0.500 mg/L	90-110	20	90-110	20	PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9407 Pore Water Generation and pH on pore water, RTI, Livonia, MI	
	Interstitial pore water pH	SM 4500-H+B	ASAP		none		0.250 mg/L	0.500 mg/L	NA	5	NA	NA		
sediment	AVS/SEM (Acid Volatile Sulphide/Simultaneously Extracted Metals)						True MDL (umoles/g)	PQL (umoles/g)						
	AVS	EPA 1629	14 days for AVS/SEM extraction, subsequent 14 days for AVS	4 oz glass jar, no headspace	none	cool, <6°C	0.00784605	0.05881000	50-150	30	50-150	30	PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9407	
	Metals						MDL (umoles/g)	PQL (umoles/g)						
	Antimony	EPA 6010	6 months				0.00149140	0.00410680	50-150	30	50-150	30		
	Arsenic						0.00227400	0.00667360	50-150	30	50-150	30		
	Cadmium						0.00022180	0.00111210	50-150	30	50-150	30		
	Chromium						0.00073300	0.00240540	50-150	30	50-150	30		
	Copper						0.00132830	0.00393420	50-150	30	50-150	30		
	Lead						0.00019500	0.00120660	50-150	30	50-150	30		
	Nickel						0.00053810	0.00425890	50-150	30	50-150	30		
	Zinc						0.00220140	0.15295200	50-150	30	50-150	30		
	Silver						0.00073280	0.00231760	50-150	30	50-150	30		
	Mercury	EPA 7471A/B	28 days				0.00001250	0.00002490	50-150	30	50-150	30		
sediment	Total Organic Carbon (TOC)	Walkley-Black	28 days	4 oz glass jar	none	cool, <6°C	113.1 mg/kg	400 mg/kg	80-120	20	80-120	20	PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9407	
	Particle size analysis	ASTM D 422-63	NA	Two 4 oz glass jar; 1 gallon ziplock bag		none	NA	NA	NA	NA	NA	NA	CQM Inc. Green Bay, WI	
	Cation Exchange Capacity (CEC)	EPA 9081	180 days			cool, <6°C	0.5 meq/100g	0.1 meq/100g	62-138	47	NA	NA	Pace -Kansas	
ground-water	Total suspended solids (TSS)	SM 2540D	7 Days	1 Liter plastic	none	cool, <6°C not frozen	0.15 mg/L	10 mg/L	80-120	20	80-120	20	PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9407	It is assumed that water samples will be field filtered for dissolved metals. T.Rec.Phenols, Pace Kansas
	Phenols Analyte						True MDL (ug/L)	PQL (ug/L)						
	2,3,4,6-Tetrachlorophenol	EPA 8270C	7 days extraction/ 40 days analysis	1 Liter glass	none		4.51	5	70-130	20	70-130	20		
	2,4,5-Trichlorophenol						0.76	5	70-130	20	65-130	22		
	2,4,6-Trichlorophenol						1.05	5	70-130	20	64-130	20		
	2,4-Dichlorophenol						1.19	5	69-130	23	60-130	27		
	2,4-Dimethylphenol						0.93	5	38-130	33	10-135	41		
	2,4-Dinitrophenol						0.86	10	38-130	38	12-162	46		
	2-Chlorophenol						1.03	5	58-130	27	47-130	41		
	2-Methylphenol(o-Cresol)						0.96	5	48-130	28	33-130	39		
	2-Nitrophenol						0.85	5	70-130	20	53-130	21		
	3&4-Methylphenol (m&p Cresol)						1.28	5	43-130	29	35-130	39		
	4,6-Dinitro-2-methylphenol						0.62	5	53-130	20	15-150	33		
	4-Chloro-3-methylphenol						1.43	5	61-130	27	51-130	33		
	4-Nitrophenol						2.23	10	18-130	32	10-130	25		
	Cresols (Total)						2.24	5	10-150	20	10-150	20		
	Pentachlorophenol						0.75	10	54-130	20	10-149	32		
	Phenol						0.54	5	28-130	20	22-130	33		
	Metals (Total and Dissolved)						MDL (ug/L)	PQL (ug/L)						
	Antimony	EPA 6020	6 months	Two 250 mL plastic	HNO3 <2pH		0.115	1.00	80-120	20	75-125	20		
	Arsenic						0.193	1.00	80-120	20	75-125	20		
	Barium						0.139	1.00	80-120	20	75-125	20		
	Cadmium						0.130	1.00	80-120	20	75-125	20		
	Chromium						0.1156	1.00	80-120	20	75-125	20		
	Copper						0.381	1.00	80-120	20	75-125	20		
	Lead						0.061	1.00	80-120	20	75-125	20		
	Nickel						0.155	1.00	80-120	20	75-125	20		
	Zinc						4.069	10.00	80-120	20	75-125	20		
	Mercury	EPA 7470A	28 days				0.100	0.200	85-115	20	85-115	20		
Benthic Invertebrates	Metals						MDL (mg/kg)	PQL (mg/kg)						
	Antimony	EPA 6020 in tissues	365 days	Ziplock type bags, double bagged with labels on both the interior and exterior bags.	None	Frozen and shipped on dry ice. Need at least 5 grams	0.006	0.10	80-120	20	75-125	20	PACE Analytical Services, Inc. Contact: Matt Burns email: Matt.Burns@pacelabs.com Phone: 732-343-3558 Green Bay Contact: Kang Khang email: kang.khang@pacelabs.com Phone: 920-321-9408	Sample mass needed for metals is 5 grams
	Arsenic						0.014	0.10	80-120	20	75-125	20		
	Barium						0.038	0.10	80-120	20	75-125	20		
	Cadmium						0.011	0.10	80-120	20	75-125	20		
	Chromium						0.0388	0.10	80-120	20	75-125	20		
	Copper						0.064	0.50	80-120	20	75-125	20		
	Lead						0.049	0.10	80-120	20	75-125	20		
	Nickel						0.018	0.10	80-120	20	75-125	20		
	Zinc						0.890	2.00	80-120	20	75-125	20		
	Mercury	EPA 7471 A/B					0.00134	0.0100	85-115	20	85-115	20		
	Soil Moisture Content	ASTM D2974-87	28 days	100g glass with tight fitting lid		0°C to 6°C	0.10%	0.10%	NA	5	NA	NA		
	Wet Weight	NA	NA	NA		NA	NA	NA	NA	NA	NA	NA		
	Dry Weight	NA					NA	NA	NA	NA	NA	NA		

Pace Notes

Laboratory performing the work is Pace Green Bay unless otherwise noted

Method Detection Limits and Reporting Limits are periodically updated and subject to change without notice.

Pace Standard QC Level IV and Pace Standard excel EDD will be provided, except for Particle Size, Water Holding Capacity, and pore water pH.

Particle Size and Water Holding Capacity will be done by CQM, Inc. Green Bay, WI

Water Holding Capacity defined as available water capacity of soil to determine efficiency of soil in crop production, analysis required include Sieve, Hydrometer, and Atterberg Limit

Particle Size assumes both sieve and hydrometer are required.

Pore Water Extraction will be done by RTI, Livonia, MI sediment to be shipped directly to RTI via ground shipment by WSP. Any overlying water on the sample when received will be considered pore water and incorporated.

Pore Water pH will be done by RTI, their QC Level IV report will be appended to Pace's. RTI's results will not be included in the EDD

AVS/SEM requires 3 week prior notice or meeting hold times is not guaranteed. All samples for this analysis must be submitted at the same time. AVS/SEM metals = Sb, As, Cd, Cr, Cu, Pb, Hg, Zn. Ba will not be included.

It is assumed that water samples will be field filtered for dissolved metals.

Attachment A – WSP SOPs

WSP Standard Operating Procedures

Table of Contents

SOP Number and Title

1 Note Taking and Log Book Entries
2 Sample Container, Preservatives, and Holding Times (Table 1)
3 Groundwater Sampling (revised July 2012)
4 Surface Water and Sediment Sampling
5 Sediment/Sludge Sampling with Ekman or Ponar Dredge
6 Sediment/Sludge Sampling Using Hand Corer
7 Sludge Sampling with Sludge Judge
8 Sludge Sampling with Coring Tube
9 Soil Sampling Using Bucket Auger
10 Split Spoon Soil Sampling (Figure 2)
11 Soil Sampling Using Hand Trowel
12 Chip, Wipe, and Sweep Sampling
13 Concrete Core Sampling
14 Waste Pile Sampling
15 Decontamination of Drilling Equipment
16 Decontamination of Submersible Pumps
17 Decontamination of Water Level Indicators
18 Decontamination of Interface Probe
19 Decontamination of Sampling Equipment
20 Sample Shipping Procedures
21 Field QA/QC Samples
22 Soil Head Space Screening (Field Procedure)
23 Underground Utility Locating
24 Soil Sampling Using GeoProbe® System or Equivalent
25 Groundwater Sampling Using GeoProbe® System or Equivalent
26 Managing Investigation Derived Waste
27 Soil Sampling Using a Rotosonic Sampler
28 Sampling of Private Water Supply Wells
29 Aquifer Pumping Tests
30 In Situ Single Well Hydraulic Conductivity Test (Slug Test)

31 Screening of Soils for Metals Using a Delta Handheld X-Ray Fluorescence Analyzer
32 Composite Soil Sampling
33 Sediment Pore Water Sample Collection Performed by Laboratory
34 Multi-Habitat Macroinvertebrate Collection Procedure

Standard Operating Procedure – 1

Note Taking and Log Book Entries

Materials:

Permanently bound log book (no spiral-bound log books)

Black or blue ballpoint pen (waterproof ink)

Procedure:

1. Use black or blue ballpoint pen with waterproof ink. Felt-tip pens should not be used.
2. Reserve the inside front cover for business cards from key personnel who visit the site (including the person in charge of the log book).
3. On the first page of the log book, place a return for reward notice, WSP's phone number, and the project manager's name.
4. Enter the following on the second page of the log book: project name, project number, project manager's name, onsite contacts, onsite telephone number and address, telephone numbers for all key personnel, and emergency fire and medical telephone numbers.
5. Number each page, initial each page, and put the date at the top of each page. Start a new page for each day. At the end of a day, summarize the day's activities, sign the page, and put a slash through the rest of the blank lines. Start the next day on a new page.
6. Enter the time (in military time, e.g., 0830) in the left column of each page when an entry is recorded in the field notebook.
7. If a mistake is made in an entry, cross out the mistake with one line and initial the end of the line.
8. At all times, maintain the chain of custody on the field log book.

Content:

1. Be sure that log book entries are LEGIBLE and contain accurate and inclusive documentation of project field activities.
2. Provide sufficient detail to enable others to reconstruct the activities observed.
3. Thoroughly describe all field activities while onsite. Be objective, factual, and thorough. Language should be free of personal feelings or other terminology that might prove inappropriate.
4. Describe problems, delays, and any unusual occurrences such as wrong equipment or breakdowns along with the resolutions and recommendations that resulted.
5. Fully document any deviations from or changes in the work plan.
6. Describe the weather and changes in the weather, particularly during sampling events.

7. Sketch a map of the facility or areas onsite where activities are occurring, especially the location of sampling points.
8. During sampling activities, record all information pertaining to the sampling event. Include descriptive locations and diagrams of the sample locations, time, sample media, analysis, sampling procedure, equipment used, sizes and types of containers, preservation and any resulting reactions, sampling identification (especially for duplicate samples), shipping procedures (record airbill numbers), and addresses.
9. Note decontamination or disposal procedures for all equipment, samples, and protective clothing and how effectively each is performed.
10. If possible, photograph all sample locations and areas of interest. Maintain a photographic log in the field log book and include:

    Date, time, photographer, name of site, general direction faced, description of the subject taken, and sequential number of the photograph and the roll number.
11. Record the names and affiliations of key personnel onsite each day.
12. List all field equipment used and record field measurements, including distances, monitoring and testing instrument readings (e.g., photoionization detector (PID), organic vapor analyzer (OVA), pH, conductivity, model numbers, etc.), and calibration activities.
13. Record proposed work schedules and changes in current schedules in the log book.
14. Describe site security measures.
15. Include drum inventory for all investigation-derived waste (IDW) materials generated during site activities. Provide information on how IDW material was labeled.

Standard Operating Procedure - 2

Sample Container, Preservatives, & Holding Times

Scope:

This operating procedure describes the ways and means of selecting the appropriate sampling containers for environmental sampling.

Application:

The purpose of this procedure is to assure that sample volumes and preservatives are sufficient for analytical services required under EPA-approved protocols.

Materials:

Sample containers

Sample container labels

Indelible (waterproof) markers or pens

Clear tape

Procedures:

1. Refer to Table 1 for minimum sample volume and glassware types required for sampling a particular matrix and compound class.
2. Select the appropriate glassware (i.e., bottles or jars) from those provided by the analytical laboratory. Verify that the analytical laboratory has provided the correct number of sample containers and the correct preservatives for the project per the sampling plan requirements.
3. The analytical laboratory should always provide extra sample containers for all analytical parameters in case of breakage or other problems encountered in the field. This is particularly true for VOC sample containers (i.e., 40-ml vials).
4. Report any discrepancies or non-receipt of specific types of sample containers to the Quality Assurance Officer immediately. Arrangements should be made with the laboratory to immediately ship the missing or additional sampling containers to the project site.
5. Apply WSP sample labels to the sample containers.
6. Information on the sample labels should contain the following data:

    Site/Project name

    Project/Task number

    Unique sample identification number

    Sample date

    Time of sample collection (military system, e.g., 0000 to 2400 hours)

Analytical parameters

Preservative

Sampling personnel

7. Once sample containers are properly labeled, the sample labels should be wrapped with clear tape to prevent deterioration of sample label.
8. Proceed with the sample collection per the sampling plan requirements.
9. Collected samples should be immediately placed in an iced cooler to maintain as close as possible a 4*C atmosphere for shipment to the analytical laboratory. Follow sample shipping procedures detailed in Sample Shipping Standard Operating Procedures.
10. Recommended order of sample collection:

    In-situ measurements (e.g., temperature, pH, specific conductance)

    Volatile organic analytes (VOA)

    Purgeable organic carbon (POC)

    Purgeable organic halogens (POX)

    Total organic halogens (TOX)

    Total organic carbon (TOC)

    Extractable organics

    Total petroleum hydrocarbons (TPH)

    Total metals

    Dissolved metals

    Microbiologicals

    Phenols

    Cyanide

    Sulfate and chloride

    Turbidity

    Nitrate and ammonia

    Radionuclides

Table 1 – Sample Containers, Preservatives, and Holding Times

Parameter	Matrix	Sample Container Size and Type	Preservatives	Holding Time
Metals, except mercury and hexavalent chromium	Solid	4-oz. glass jar	Cool to <6° C	180 days
Mercury	Solid	4-oz. glass jar	Cool to <6° C	28 days
Hexavalent chromium	Solid	4-oz. glass jar	Cool to <6° C	24 hours
Metals, except mercury and hexavalent chromium	Aqueous	500-ml plastic container with Teflon-lined plastic cap	HNO3, pH<2 Cool to <6° C	180 days
Mercury	Aqueous	500-ml plastic container with Teflon-lined plastic cap	HNO3, pH<2 Cool to <6° C	28 days
pH	Solid	Three 4oz glass jar with Teflon-lined cap; 1 Gallon zip lock bags	Cool to <6°C	ASAP
Total Organic Carbon	Solid	Three 4oz glass jar with Teflon-lined cap; 1 Gallon zip lock bags	Cool to <6°C	28 days
Water Holding Capacity	Solid	Three 4oz glass jar with Teflon-lined cap; 1 Gallon zip lock bags	Cool to <6°C	N/A
Cation Exchange Capacity	Solid	Three 4oz glass jar with Teflon-lined cap; 1 Gallon zip lock bags	Cool to <6°C	180 days

Parameter	Matrix	Sample Container Size and Type	Preservatives	Holding Time
Particle Size Analysis	Solid	Three 4oz glass jar with Teflon-lined cap; 1 Gallon zip lock bags	Cool to <6°C	N/A
14-Day Earthworm survival & Biomass Toxicity Test	Solid	plastic of PTFE containers 2 L needed	Cool to 4°C; store in dark until testing	72 hours
Lead	Solid	Three 4oz glass jar with Teflon-lined cap	Cool to <6°C	N/A
P. promelas and C.dubia chronic toxicity test	Aqueous	5 gal plastic container; no headspace	Cool to 4°C; store in dark until testing	N/A
pH	Aqueous	Two 250 mL plastic	Cool to <6°C	1 none; 1 HNO3<2 pH
Hardness	Aqueous	Two 250 mL plastic	Cool to <6°C	1 none; 1 HNO3<2 pH
Total Recoverable Phenols	Solid	Three 4oz glass jar with Teflon-lined cap	Cool to <6°C	N/A
H. Azteca 28-Day Survival and Growth C. dilates 10-Day toxicity test	Solid	plastic or PTFE containers; can use 1L or gal; need 5L of sample	Cool to 4°C; no headspace; store in dark until testing	2 weeks; up to 8 weeks under nitrogen
Interstitial pore water Ammonia	Solid	3 gallon covered plastic bucket	Cool to 4°C	14 days
AVS/SEM	Aqueous	4oz glass jar; no headspace	None	14 days
Total Suspended Solids	Aqueous	1L plastic bottle	none	7 days
Total phenols	Aqueous	1L plastic bottle	H2SO4 <2 pH	28 days
Metals	Benthic	5 grams in plastic bags	Frozen, ship on dry ice	N/A

Standard Operating Procedure - 3

Groundwater Sampling Procedures

1.1.1.1 Scope and Application

This groundwater sampling procedure is designed to ensure that collected groundwater samples will be representative of groundwater present in the aquifer or target formation and that the groundwater samples have not been altered or contaminated by the sampling and handling procedures. These procedures can be applied to permanently, or temporarily, installed monitoring wells, temporary wells constructed using "direct-push" techniques, wells with installed plumbing, remedial groundwater treatment systems, and excavations where groundwater is present. The topics detailed in this Standard Operating Procedure (SOP) include equipment and supply selection, equipment construction materials, and purging and sampling techniques.

1.1.1.2 Acronym List

bgs	below ground surface
C	Celsius
CID	casing inside diameter
DI	deionized
DNAPL	dense non-aqueous phase liquid
DO	dissolved oxygen
DTW	depth to water
Eh	redox potential
HASP	Health and Safety Plan
L/min	liters per minute
LNAPL	light non-aqueous phase liquid
mg/l	milligrams per liter
mV	millivolts
NAPL	non-aqueous phase liquid
NTU	Nephelometric Turbidity Unit
ORP	oxygen reduction potential
pH	hydrogen ion activity
PID	Photoionization Detector
PPE	Personal Protective Equipment
PVC	polyvinyl chloride
QAPP	Quality Assurance Project Plan

SOP	Standard Operating Procedure
SU	Standard Units
TD	total depth
TOC	top of casing
µm	micron
µS/cm	microsiemens per centimeter
VOCs	volatile organic compounds

Materials

- Field notebook
- Personal Protective Equipment (PPE)
- Groundwater monitoring data log forms
- Well key(s), as needed
- Adjustable wrench or manhole wrench
- Plastic sheeting
- Air quality monitoring equipment (e.g., photoionization detector [PID]), as needed
- Flashlight or mirror
- Electronic water level indicator or interface probe
- Pump or bailers, tubing, and associated lanyard materials
- Water quality meter(s) with calibration reagents and standards
- Field test kits, as needed
- Pocket knife or scissors
- Deionized (DI) water
- Power supply, as needed
- Buckets or drum(s) for water storage
- Sample bottles, labels, indelible markers, and clear tape

1.1.1.3 Compulsory Prerequisites and Background Information

Before conducting field activities, review the site Health and Safety Plan (HASP) and determine the proper level of PPE. The reader is encouraged to read this entire SOP before beginning any onsite activities. This SOP is designed to provide the user with a general outline for conducting groundwater sampling and assumes the user is familiar with basic field procedures, such as recording field notes, utility location, sample shipment procedures, sample collection and quality assurance procedures, equipment decontamination, and investigation derived waste management procedures. This SOP does not cover the development of a sampling and analysis plan, nor does it cover the selection of analytical

procedures, or evaluation of the analytical results. These topics require a significant amount of planning and are more appropriately addressed in a site-specific work plan.

Before sampling, be sure to review the project specific work plan or Quality Assurance Project Plan (QAPP) and any applicable state and federal guidelines or sampling procedures. Groundwater samples may be collected from a number of different configurations. Each configuration is associated with a unique set of sampling equipment requirements and techniques. As possible, monitoring wells or borings should be sampled by starting with the upgradient (or clean locations) and proceeding downgradient (in the order from least to most contaminated locations) for the remaining monitoring wells or borings.

1.1.1.4 Equipment Selection Considerations

Use groundwater purging and sampling equipment constructed of only non-reactive, non-leachable materials that are compatible with the environment and the selected analytes. Additional supplies such as reagents, preservatives, and field measurement equipment are often necessary.

Pump Selection

Groundwater sampling is accomplished by using in-place plumbing and dedicated pumps or by using portable pumps/equipment when dedicated systems are not present. In selecting groundwater purging and sampling equipment, give consideration to:

- the depth of the well
- the depth to groundwater
- the volume of water to be evacuated
- the sampling and purging technique
- the analytes of interest

Sampling equipment will usually consist of peristaltic pumps, variable speed electric submersible pumps, bladder pumps, inertial lift pumps, or bailers.

- Restrictions and Precautions:
- Follow all manufacturer's instructions for assembly, operation, and maintenance specific to your equipment
- For wells where the water level is below the limit of suction (approximately 25 feet to 30 feet below ground surface, bgs), and/or where there is a large volume of water to be purged, a peristaltic pump should not be used
- Purging and sampling with an inertial lift pump (e.g., polyethylene tubing with a bottom check valve) are performed by oscillating the tubing at the intake depth to drive a column of water to the surface. Inertial lift pumps cause a surging action that may cause increased turbidity, loss of volatiles, aeration, and degassing of samples. These pumps can be used when collecting non-sensitive samples; however, should not be used for volatile organic compounds (VOCs) or gas-sensitive samples. A peristaltic pump may be attached to the sample tubing and used to pump water to the surface

- VOCs and extractable organics should not be sampled with a peristaltic pump unless a vacuum jug or gravity method is employed and the method was previously approved.
- Bailers may also be used for purging in appropriate situations; however, their use is discouraged by many programs. Because bailers have been shown to increase turbidity while purging and sampling, bailers should be avoided when sampling for trace element, metal, polychlorinated biphenyls, and pesticide constituents. Bailers, if improperly used, may also strip VOCs from the water column being sampled
- Bailers with a controlled flow bottom when should be used when collecting volatile organic samples
- Before use, install a check valve at the end of the purge tubing to prevent backflow
- Pump housing and tubing parts must be compatible with the analytes of interest. Parts can be composed of various materials, usually polyvinyl chloride (PVC), Teflon, polyethylene, polypropylene, and stainless steel, or other non-inert materials; be sure to use non-metallic or stainless steel tubing if sampling for metals
- Electrical cords and any cabling must be sealed in Teflon, polyethylene, or polypropylene, or be stainless steel
- Lanyard material must be non-reactive, non-leachable material (e.g., cotton twine, nylon, stainless steel, Teflon coated, polyethylene, or polypropylene; stainless steel, Teflon coated, polyethylene and polypropylene lanyards may be decontaminated for future use

1.1.1.5 Groundwater Indicator Parameter Monitoring Equipment

WSP regularly conducts field testing for the following in-situ or field-collected parameters: groundwater level elevation, hydrogen ion activity (pH), specific conductance (conductivity), temperature, dissolved oxygen (DO), oxygen reduction potential (ORP), and turbidity. Field testing is usually conducted due to the unstable nature of these parameters; laboratory determinations will likely not be representative of field conditions.

1.1.2 pH

pH is a measure of the effective concentration (or activity) of hydrogen ions and is expressed as the negative base-10 logarithm of the hydrogen-ion activity in moles per liter. Uncontaminated groundwater typically exhibits a pH ranging from 5 to 9 Standard Units (SU). Changes in pH from background may indicate the presence of groundwater contamination or that existing contamination has spread and can be very useful in identifying well construction or maintenance problems.

1.1.3 Specific conductance

Specific conductance, or conductivity, measures the ability of water to conduct an electric current. It is generally reported in microsiemens per centimeter (µS/cm), as natural waters commonly exhibit specific conductance well below 1 µS/cm. Total dissolved solid concentrations may be approximated from specific conductance data; high readings may indicate contamination, especially if the readings are elevated compared to background. Alternatively, elevated specific conductance may indicate inadequate well development, grout contamination, or an inadequate grout seal.

1.1.4 Temperature

Temperature is not necessarily an indicator of groundwater chemical stabilization, and is generally not very sensitive in distinguishing between stagnant casing water and formation water. Nevertheless, temperature is important for data interpretation. For example, stabilized temperature readings that are representative of typical groundwater conditions help demonstrate that the sample was collected in a manner that minimized exposure to elevated temperature variations, e.g., heating from the electric motor of a submersible pump. Elevating the temperature of a sample may result in loss of VOCs or the progression of chemical reactions that may alter the sample quality in an undesirable manner.

1.1.5 DO

DO has been demonstrated to be a reliable indicator of the chemical stabilization of purge water under most groundwater purging and sampling circumstances. Concentrations of DO in groundwater generally range from 1 to 4 milligrams per liter (mg/l) and should only be measured with a flow-through cell. Relatively low DO concentrations (< 1 mg/l) in groundwater may indicate the biodegradation of organic contaminants, including VOCs. DO is a good indicator when sampling for VOCs, because erratic or elevated DO readings may reflect procedures that are causing excessive agitation and aeration of the ground water being drawn from the well and subsequent loss of VOCs. Artificially aerated groundwater may also adversely affect dissolved metals analyses.

1.1.6 ORP

ORP is a numerical index of the intensity of the oxidizing or reducing conditions within an aqueous solution such as ground water. Oxidizing conditions are indicated by positive potentials and reducing conditions are indicated by negative potentials. ORP measurements are generally expressed in millivolts (mV). The ORP of natural (uncontaminated) groundwater typically ranges from +500 to -100 mV. Groundwater contaminated with organic compounds generally exhibits depressed ORP values compared to background conditions and may exhibit ORP values as low as -400 mV. ORP must be measured with a flow-through cell and may not be an appropriate stabilization parameter for some groundwater conditions.

1.1.7 Turbidity

Turbidity, which is the visible presence of suspended mineral and organic particles in a ground water sample, can be useful to measure during purging. Relatively high or erratic measurements may indicate inadequate well construction, development or improper sampling procedures, such as purging at an excessive rate that exceeds the well yield. Purging and sampling in a manner that produces low-turbidity water is particularly important when analyzing for total metals, which may exhibit artificially elevated concentrations in high-turbidity samples. Generally, the turbidity of in-situ groundwater is very low (at or below 10 Nephelometric Turbidity Units, NTUs); however, some groundwater zones may have natural turbidity higher than 10 NTUs.

The groundwater indicator parameters selected for field testing should be determined by the project team. Field testing of groundwater indicator parameters should be conducted with cleaned, calibrated equipment. The following procedures should be followed and all observations and measurements recorded in the field book.

- Select water quality monitoring equipment that meets the project's data quality objectives and applicable regulatory requirements for field testing measurements
- Record manufacturer name, model number, and identifying number such as a serial number for each instrument
- Follow the manufacturer's instructions for assembly, operation, calibration, and maintenance specific to your equipment
- Ensure that all equipment is in proper working condition, not damaged, and that batteries are properly charged before using the equipment for field testing measurements
- Calibration of instruments should occur in the field, as close to the time of use as possible and, at least, be at the frequency suggested by the manufacturer
- Although water quality monitoring equipment may vary in configuration or operation, project specific calibration requirements must be met
- Document the time, date, and instrument reading of acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements
- Document the date of receipt, expiration date, date of first use for all standards and reagents, grade and concentration (or other value) for the standard in the appropriate measurement units
- Thoroughly rinse the instrument with DI water and fresh standards or reagents when calibrating or verifying the calibration or when taking sample measurements. For in-situ measurements, ensure adequate flushing of the instrument with fresh sample water prior to taking measurements. Residual remaining on the instrument may affect the measurement
- If calibration fails to meet criteria, follow the manufacturer's instructions for corrective action to correct instrument performance; record discontinuation of equipment due to non-compliance with calibration failure

1.1.7.1 Field Measurements of Groundwater Indicator Parameters

Indicator parameters are measured in the field to evaluate well stabilization during purging, provide information on general ground water quality, evaluate well construction, or indicate when well maintenance is needed. Indicator parameter data also may be helpful in evaluating the presence of groundwater contamination.

After calibrating the groundwater quality monitoring equipment, follow the manufacturer's instructions to select the display for readings the necessary geochemical parameters. Field testing measurements should be collected in accordance with the purge and sampling schedules provided in Sections 9.8 and 9.9. The following procedures should be followed and all observations and measurements recorded in the field book.

- Collection of field parameters should be completed within 15 minutes of sample collection
- Immerse the probe in a flow-through cell, sample cup or beaker, or at the desired depth in the well or borehole, and wait for stabilization of the reading before recording the measurement
- If collecting field measurements ex-situ, agitate or swirl the sample during the stabilization period.

- Specific conductance is sensitive to temperature. If the meter is equipped with manual temperature compensation, adjust the conductivity meter to the water temperature per manufacturer's instructions. If the water temperature varies significantly from 25°Celcius (C), measure the temperature with a calibrated device, record the temperature, and correct for temperature according to the manufacturer's specifications
- ORP meters may present redox potential (Eh) values; depending on the desired results, conversion between ORP and Eh may be necessary
- **DO and ORP readings must be obtained in a manner in which the sample is not exposed to air prior to the measurement (e.g., flow-through cell).**

Record all field-testing measurement data, to include the following:

- Project name and location
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

1.1.7.2 Set-Up Procedures

Once the equipment and supplies have been selected, gathered, and calibrated, prepare for groundwater sampling. As the following steps are completed, note all observations and measurements on the groundwater monitoring data log.

- Verify locations of wells or borings, media to be sampled, and analytes
- Record the approximate ambient air temperature, precipitation, wind, tidal conditions, and other field conditions the field book. In addition, any site-specific conditions or situations that could potentially alter the ground water samples or water level measurements should be recorded
- Inspect well for soundness of protective casing and surface ground seal
- Survey around the base of the well and wellhead with a PID, as necessary
- Remove the well cover and all standing water around the top of the well casing before opening the well cap
- New plastic sheeting should be placed on the ground surface around the well or boring to prevent contamination of the pumps, hoses, lanyards, etc., in the event they accidentally come into contact with the ground surface or, for some reason, they need to be placed on the ground during the purging event; keep the plastic as clean as possible and replace as necessary
- Unlock and carefully remove well cap, if present; allow the groundwater level to equilibrate after removing the cap before conducting any downhole testing or measurements

- Survey well casing with a PID, as necessary
- Survey breathing zone to ensure that the level of PPE is appropriate
- Position fuel powered equipment downwind and at least 10 feet from the well head or boring. Make sure that the exhaust faces downwind

1.1.7.3 Groundwater Level and Depth Measurement Procedures

The measurement of the groundwater level in a well is frequently conducted in conjunction with groundwater sampling to determine the "free" water surface. This potentiometric surface measurement can be used to establish groundwater direction and gradients. Groundwater level and well depth measurements are needed to determine the volume of water in the well casing prior to purging the well for sampling purposes. The following procedures should be followed and all observations and measurements recorded in the field book.

- Measure and record the height of the top of the well riser above the ground; if well is a flush mount, measure and record the top of the well riser below the ground surface
- Measure the casing inside diameter (CID) and record in inches
- From the top of the casing (TOC) at the surveyor's mark, if present, measure the depth to water (DTW) to the nearest 0.01-foot with an electronic water level indicator (record in feet below TOC); if no mark is present, mark a location with a metal file or indelible marker on the north side of the casing for future reference
  - All DTW measurements must be made and recorded to the nearest 0.01 foot
  - Water level measurements from boreholes, piezometers, or monitoring wells used to define the water table or a single potentiometric surface should be collected within less than 24 hours. In certain situations, water level measurements should be made within a shorter interval, if possible:
  - Aquifers influenced by tides, recent precipitation, barometric pressure, river stage, bank storage, impoundments and/or unlined ditches, intermittent pumping of production, irrigation, or supply wells
- Total depth (TD) of the well or boring should not be conducted immediately before purging and sampling; as possible, delay purging and sampling activities for at least 24 hours after TD measurement or for a time sufficient to meet the purge stabilization criterion for turbidity. Alternately, measure TD after sample collection
  - All TD measurements must be made and recorded to the nearest 0.1 foot
  - Because of tape buoyancy and weight effects encountered in deep wells with long water columns, it may be difficult to determine when the tape end is touching the bottom of the well. Sediment in the bottom of the well can also make it difficult to determine total depth. Care must be taken in these situations to ensure accurate measurements
  - As a cautionary note, when measuring TD with an electronic water level indicator, measure and add the length of the probe beneath the circuit closing electrodes to the depth measured to obtain the true TD
- Inspect water surface in the well or boring; use flashlight if necessary. Note any observable floating product (light non-aqueous phase liquids; LNAPLs) and sinking free product layer (dense non-

aqueous phase liquids; DNAPLs). Measure the thickness of the LNAPL and/or DNAPL layer using an appropriate interface gauging probe or a weighted tape coated with the appropriate reactive indicator paste for the suspected NAPL. DO NOT PURGE OR SAMPLE GROUNDWATER IN A WELL CONTAINING LNAPL. NAPL sampling is discussed in Section 9.9

1.1.7.4 Groundwater Purging

Purging is the process of removing stagnant water from a well, immediately prior to sampling, causing its replacement by groundwater from the adjacent formation that is representative of actual aquifer conditions.

The selection of the purging technique and equipment is dependent on the hydrogeologic properties of the aquifer, especially depth to groundwater and hydraulic conductivity. The intent of proper purging is to stabilize the groundwater level and minimize the hydraulic stress to the hydrogeologic formation. Every attempt must be made to match the pumping rate with the recharge rate of the well before evaluating the purging completion criteria.

In order to determine when a well has been adequately purged, geochemical parameters should be monitored, at a minimum, the pH, specific conductance and turbidity of the groundwater removed during purging and, in the case of permanent monitoring wells, the volume of water removed should be observed and recorded. Document and report the following, as applicable:

- Purging rate
- Drawdown in the well, if any
- Pump or tubing intake placement.
- Length and location of the screened interval

There are several purging strategies that may be used, depending on specific conditions encountered for given sampling situations. Purging options detailed in this SOP are:

- Traditional Multiple Volume Purge
- Low-Flow Purge
- Temporary Well Purge
- Private Water Well or In-Place Plumbing Purge

No-flow groundwater sampling techniques are discussed in Section 9.8.

1.1.7.5 Multiple Volume or 'Traditional' Purge

A multiple volume or 'traditional' purge involves removing a minimum of three well volumes of water before sample collection. Generally, an adequate purge is achieved when three to five volumes have been removed. The following procedures should be followed and all observations and measurements recorded in the field book.

- Calculate the volume of water in a well or boring using the following equation
- Volume (in gallons) = (TD – DTW)*CID2*0.041; where:
- TD = total depth (feet)

- DTW = depth to water (feet)
- CID = casing inner diameter (inches)
- Alternately, the volume of water in a well or boring may also be calculated by multiplying the water column height by the gallons per foot of water for the appropriate well or boring diameter:

CID	Gallons per foot of water	Gallons per foot, three water columns
0.75-inch	0.02	0.06
1-inch	0.04	0.12
1.25-inch	0.06	0.18
2-inch	0.16	0.48
3-inch	0.37	1.11
4-inch	0.65	1.98
5-inch	1.02	3.06
6-inch	1.47	4.41
12-inch	5.88	17.61

- Calculate the total volume of the pump, associated tubing and container for in-situ measurements (flow-through cell), if used, using the following equation:

  Volume (in gallons) = P + ((0.0041)*D2*L) + fc; where:

  P = volume of pump (gallons)

  D = tubing diameter (inches)

  L = length of tubing (feet)

  fc = volume of flow-through cell (gallons)
- Install the pump or tubing to the depth prescribed in the work plan or QAPP, as dictated by the purge and sampling method
  - for wells with a completely submerged screen or if a bailer is being used, the pump, tubing or bailer should be placed above the screen at the top of the water column
  - for wells with a partially submerged screen, the pump or tubing should be placed within the middle of the saturated portion of the screened interval
- Prepare for the collection of purge water into an appropriate storage container
- Begin purging at a rate that will not cause excessive turbulence in the well; commonly less than 1 gallon per minute. Stabilization parameters should be collected every 0.5 to 1 well volume, or more frequently, record field measurements and observations of water color, suspended particulates,

discoloration of casing, casing diameter and material, any unusual occurrences during sampling, and any pertinent weather details in the field book

- The minimum volume removed must be more than the volume of the pump and sampling tube capacity and the flow through cell (if used). After the minimum purge volume has been removed, review the geochemical measurements to ensure that readings have stabilized. Stabilization occurs when at least three consecutive measurements are within:

Traditional Purge Stabilization Parameters	
pH	± 0.1 SU
Specific Conductance	± 3%
Temperature	± 3%
DO	± 0.2 mg/l or 10% (flow-through cell only)
Turbidity	± 10% for values greater than 1 NTU
ORP	± 10 millivolts (flow-through cell only)

- If the geochemical measurements have not stabilized within five volumes, it is at the discretion of the project team whether or not to collect a sample or to continue purging. If, after five well volumes, pH and conductivity have stabilized and the turbidity is still decreasing and approaching an acceptable level, additional purging should be considered to obtain the best sample possible, with respect to turbidity
- If drawdown is observed on initiation of pumping, reduce the pump speed and attempt to match the drawdown of the well. If the drawdown stabilizes, maintain that level, however, if it continues to lower, "chase" the water column until the well is evacuated
- If the well or borehole is purged dry before removing three well volumes, allow well or boring to recharge (commonly 90%) and proceed immediately to sample collection. If adequate sampling volume is available immediately upon completion of purging, the well should be sampled immediately. If recovery exceeds 2 hours, sample as soon as sufficient volume is available, or within 3 hours of purging. If possible, sampling of wells which have a slow recovery should be scheduled so that they can be purged and sampled in the same day, after adequate volume has recovered
- Once purging is complete, proceed to sample collection, as described in Section 9.9

Low-Flow Purge

Low-flow purging is generally used in low permeability units, when calculated purge volumes for traditional purging are excessive, or when vertical characterization within a screened interval is desired.

In-line flow-through cells equipped with water quality meters with continuous readout displays are highly recommended. The following procedures should be followed and all observations and measurements recorded in the field book.

- Calculate the total volume of the pump, associated tubing and container for in-situ measurements (flow-through cell), if used, using the following equation:

  Volume (in gallons) = P + ((0.0041)*D2*L) + fc; where:

  P = volume of pump (gallons)

  D = tubing diameter (inches)

  L = length of tubing (feet)

  fc = volume of flow-through cell (gallons)

- Install the pump or tubing to the depth prescribed in the work plan or QAPP, as dictated by the purge and sampling method
  - for wells with a completely submerged screen, the open or screened interval is less than 10 feet, and the aquifer recovery rate is approximately equal to the purge rate, the intake should be placed in the middle of the screen or open interval
  - for wells with a partially submerged screen, the pump or tubing should be placed within the middle of the saturated portion of the screened interval
- Prepare for the collection of purge water into an appropriate storage container
- The pump should be started at the lowest flow volume, and adjusted higher as long as the maximum drawdown is not exceeded. Purging should not exceed 0.1 to 0.5 liters per minute (L/min)
- Water level should optimally be monitored continuously, but at a minimum, at 30 seconds to 5 minutes intervals (depending on the hydraulic conductivity of the aquifer, diameter of the well, and pumping rate) during purging. Ideally, a steady flow rate should be maintained that results in a stabilized water level (less than 0.3 feet of variation). Pumping rates should, if needed, be reduced to the minimum capabilities of the pump to ensure stabilization of the water level. However, care should be taken to maintain pump suction and to avoid entrainment of air in the tubing. Record each adjustment made to the pumping rate and the water level measured immediately after each adjustment.
- If the recharge rate of the well is very low, care should be taken to avoid loss of pressure in the tubing line, cascading through the sand pack, or pumping the well dry. In these cases, purging should be interrupted before the water in the well reaches a level below the top of the pump. Sampling should commence as soon as the volume in the well has recovered sufficiently to permit collection of samples.
- During purging, monitor and record geochemical parameters at 30 seconds to 5 minutes intervals. Stabilization occurs once at least three equipment volumes have been purged and the following criteria have been met over three successive measurements made at least three minutes apart:

Low-Flow Purge Stabilization Parameters	
Water Level Drawdown	<0.3 feet
pH	± 0.1 SU
Specific Conductance	± 3%
Temperature	± 3%
DO	± 0.2 mg/l or 10% (flow-through cell only)
Turbidity	± 10% for values greater than 1 NTU
ORP	± 10 millivolts (flow-through cell only)

- Once purging is complete, proceed to sample collection, as described in Section 9.9.

1.1.7.6 Temporary Well Purge

Procedures used to purge temporary ground water monitoring wells differ from permanent wells because temporary wells are installed for immediate sample acquisition. Wells of this type may include open bedrock boreholes or standard well screen and riser placed in boreholes created by hand augering, power augering, or by drilling. They may also consist of a rigid rod and screen that is pushed, driven, or hammered into place to the desired sampling interval, such as a direct push Wellpoint®, a Geoprobe® Screen Point 15/16 sampler or a Hydropunch® sampler. As such, the efforts to remove several volumes of water to replace stagnant water do not necessarily apply because stagnant water is not present. However, exposed probes that are driven through the soil to the desired water sample depth must be purged of a minimum of three probe-rod volumes of water before sampling is conducted. The longer a temporary well is in place and not sampled, the more stagnant the water column becomes and the more appropriate it becomes to apply, to the extent possible, monitoring well purging criteria to re-achieve aquifer conditions.

In cases where the temporary well is to be sampled immediately after installation, purging is conducted primarily to mitigate the impacts of installation. In most cases, temporary well installation procedures disturb the existing aquifer conditions, resulting primarily in increased turbidity. Therefore, the goal of purging is to reduce the turbidity and remove the volume of water in the area directly impacted by the installation procedure. Low turbidity conditions in these types of wells that are completed within the limit of suction are typically and routinely achieved by the use of low-flow/low stress purging techniques using variable speed peristaltic pumps.

Once purging is complete, proceed to sample collection, as described in Section 9.9.

1.1.7.7 Private Water Well or In-Place Plumbing Purge

This procedure describes the method for purging domestic or other private water supply wells with in-place plumbing. The following procedures should be followed and all observations and measurements recorded in the field book.

- Obtain information regarding the exact well location, well construction details, static water depth, well depth, casing diameter, well yield, date drilled and drilling company, previous testing, presence of water treatment components, resource aquifer, and well use. Locate the well on a map for future reference
- The sampling point should be as close to the well as possible, and before the water passes through any treatment systems, heating unit, or storage system. If available, identify the cold water tap or spigot for purging and sampling
- If a suitable sampling point is not available, try to find another well at the site or take steps to minimize the impact of any potential interferences to the sample. Document in the field notebook whether you can or cannot collect a water sample before any treatment or storage components.
- If purging from a tap or faucet, try to remove any aerators, filters, or other devices from the tap before purging
- Start to purge the system by running water from a tap or outside spigot. Observe and record the purge rate for the system. Ensure that sufficient volume is pumped to allow for the complete exchange of water into equipment, holding tanks, etc.
- The minimum volume removed must be more than the volume of the pump, holding tanks, conveyance piping, etc., and the flow through cell (if used). After the minimum purge volume has been removed, review the geochemical measurements to ensure that readings have stabilized. Stabilization occurs when at least three consecutive measurements are within:

Stabilization Parameters	
pH	± 0.1 SU
Specific Conductance	± 3%
Temperature	± 3%
DO	± 0.2 mg/l or 10% (flow-through cell only)
Turbidity	± 10% for values greater than 1 NTU
ORP	± 10 millivolts (flow-through cell only)

- Once purging is complete, proceed to sample collection, as described in Section 9.

1.1.7.8 Groundwater Sample Collection

Sampling is the process of obtaining, containerizing, and preserving (if required) a groundwater sample after the purging process is complete.

- Once purging is complete, field measurements performed to fulfill regulatory requirements, beyond those used to measure for stabilization, should be obtained after purging and before samples are collected for analysis
- While collecting samples, reduce the water flow to a thin stream. The flow rate should be high enough to deliver a smooth stream of water without splashing but low enough to permit filling of the sample bottles without turning the valve down during sampling; the sampling pump should be operated at a maximum rate of 0.25 L/min or the rate of the purging activities to avoid agitating the water; if using a bailer, lower the bailer slowly to avoid agitating the water
- Field samples must be directly transferred from the sampling equipment to the container that has been specifically prepared for that given parameter; intermediate containers should be avoided.
- Groundwater samples should be collected in the order of the volatilization (highest ability to volatilize to the lowest). Sample for VOCs first at a rate less than 0.1 L/min, taking care to remove all air bubbles from the vial and minimize agitation
- Collect remaining organic samples then inorganic samples in the following order of volatilization sensitivity:
  - VOCs
  - Purgeable organic carbon
  - Purgeable organic halogens
  - Total organic halogens
  - Total organic carbon
  - Extractable organics
  - Total metals
  - Dissolved metals
  - Phenols
  - Cyanide
  - Sulfate and chloride
  - Nitrate and ammonia
  - Radionuclides
- If submitting samples for dissolved metals analyses or conducting field testing that use filtered samples, the sample may be field filtered (Section 9.9.1)
- Affix a sample tag or label to each sample container and complete all required information (sample number, date, time, sampler's initials, analysis, preservatives, place of collection)
- Place clear tape over the tag or label
- Samples for temperature-sensitive parameters should be preserved immediately after collection by placement into an insulated cooler maintained at a temperature of approximately 4°C
- Record sample designation, date, time, and the sampler's initials on the sample tracking form and in the field book

- Complete chain-of-custody forms with appropriate sampling information
- Complete sample packing and shipping in accordance with proper procedures

1.1.7.9 Groundwater Filtration Procedures

Filtered groundwater samples are sometimes used for field kit analyses and should only be collected for laboratory use after approval from the appropriate agency and/or project manager. If filtration is necessary, the following procedures should be followed and all observations and measurements recorded in the field book.

- Use a variable speed peristaltic, bladder, or submersible pump with the in-line filter fitted on the outlet end; pressurized bailers could also be used
- At the pump discharge end, attach a clean 0.45-micron (□m) filter (for organics) or 0.1-µm (for inorganics), or appropriate sized filter, to the tubing
- Turn on the pump to a rate less than 100 mL/min, hold the filter upright with the inlet and outlet in the vertical position and pump groundwater through the filter until all atmospheric oxygen has been removed and the minimum volume of water has been flushed through the filter, in accordance with the manufacturer's specifications
- Collect the filtered samples directly into the sample container from the pump-filter assembly
- If sediment is visible in the sample container after filtration, filter break-through has occurred and the sampling and filtering process should be repeated
- Disassemble the pump head and discard the tubing and filter appropriately

1.1.7.10 No-Flow Passive Sampling Techniques

A number of alternate sampling devices are becoming available, including passive diffusion samplers, equilibrated grab samplers, and other in-situ sampling devices. These devices may be particularly useful to sampling low permeability geologic materials, assuming the device is made of materials compatible with the analytical parameters, meet data quality objectives, and have been properly evaluated. However, the site investigator should ensure the diffusion membrane materials are selected for the contaminants of concern present at the site. Comparison tests with an approved sampling method and diffusion samplers should be completed to confirm that the method is suitable for the site. Manufacturer's specifications should be strictly followed for deployment and use of these samplers.

Equilibration time for diffusion samplers depends on the time required for the environment disturbed by deployment of the sampler to return to ambient conditions and time required by the sampler to equilibrate with the ambient water. To account for this, diffusion samplers are generally deployed a minimum of 14 days prior to sample collection. Though the sampler may be deployed for an extended period (e.g., three months or longer); the analytical results will be the average of the sample equilibration time for the analyte of concern (generally the last 1 to 4 days).

No-flow grab samplers are placed in the well before sampling and remain closed. The water is then collected when the sampler is activated. Samples are either transferred to containers at the well head or the sampler is shipped to the laboratory for analysis. Examples of equilibrated grab samples include Hydrosleeve®, Snap SamplerTM, and Kemmerer Sampler.

1.1.7.11 Non-Aqueous Phase Liquid Sampling

Non-aqueous phase liquid (NAPL) is normally sampled for two reasons:

- Documentation for its existence and thickness
- Determination of the type of product so that the proper analyses can be performed to determine extent. This is only feasible for relatively recent releases as it may not be possible to identify weathered product

Disposable plastic (acrylic, clear PVC) bailers are recommended for sampling. Disposable polyethylene and polypropylene bailers are also acceptable. If a NAPL is identified in a monitoring well during the water level measurement, measure its thickness in the well.

- If the thickness of the NAPL is greater than 0.01 foot or product globules are present, collect a sample using a precleaned disposable bailer by slowly lowering a bailer and allowing the bottom to sink to 1 foot below the water surface to capture LNAPL only
- Measure the product thickness to the nearest 0.01 foot after withdrawing the bailer
- Pour a portion of the product into a glass sample container
- Follow proper sampling, packing, and shipping procedures

Before DNAPL sampling, to the extent practical, remove the full thickness of the LNAPL

- Record the quantity of LNAPL removed in the field book
- Use a peristaltic pump, inertial pump, double check valve bailer, or polyethylene tubing equipped with a bottom check valve for DNAPL sample collection
- Slowly lower the sampling equipment to the well bottom and then raise it slowly, causing as little agitation as possible
- Minimize contact of the bailer with the well casing as it is raised
- Pour a portion of the product into a glass sample container; use the bottom check valve of the bailer for transfer
- Follow proper sampling, packing, and shipping procedures

1.1.7.12 Closing Notes

At the conclusion of groundwater sampling, be sure to:

- Maintain the record of all purging and sampling observations and measurements
- Remove all temporary equipment from the well or borehole
- Decontaminate equipment and supplies
- Properly manage and dispose of all investigation derived waste

Groundwater Monitoring Data Log

Well No./Designation _____ Date: _

Site Data

Site Name: _____ WSP Sampling Team _

Site Address: _____ WSP project No.: _____

Weather Conditions: __

Well Description

Well Location: _

Well Security: _

Casing Material: Inner _____ Outer _

Organic Vapors (PID, OVA, TIP): Wellhead ppm

Breathing Zone ppm

Nonaqueous Phase (thickness):

Reference Point (e.g., top of PVC casing):

Purge Data

Purge Method:

(Note: Allow water level to equilibrate after removing well cap)

Total Well Depth (TD): _____ ft Depth to Water (DTW):

Casing Inner Diameter (CID): _____ inches

To calculate well volume: Well Vol.(gal)=$(CID)^2(0.04)(TD-DTW)$

Well Volume: _____ gal x 3=Purge Volume _____ gal

Purge Time: Begin _____ End _____

Prepurge Data: Temp _____ pH _____ Spec. Cond._____Turb. _____Other _

Volume 1: Temp _____ pH _____ Spec. Cond._____Turb. _____Other _

Volume 2: Temp _____ pH _____ Spec. Cond._____Turb. _____Other _

Volume 3: Temp _____ pH _____ Spec. Cond._____Turb. _____Other _

Volume 4: Temp _____ pH _____ Spec. Cond._____Turb. _____Other _

Volume 5: Temp _____ pH _____ Spec. Cond._____Turb. _____Other _

Volume Purged: _____ Purged Dry: Yes No

Disposal Method for Purgewater:

Water Description

Odor: Prepurge _____ Postpurge _

Color: Prepurge _____ Postpurge _

Sampling Data

Sampling Method: ____

Sampling Time: Begin _____ End ___
Analytical Parameters (circle appropriate parameters):

VOCs BNA BNE Total (Unfiltered) Metals

Dissolved (Filtered) Metals TPH PCB Cyanide

Other: __

Comments: ___

Standard Operating Procedure - 4

Surface Water and Sediment Sampling (using hand trowel)

Surface Water Sampling

Materials:

Nitrile gloves

Saranex or Tyvek suit

Vinyl gloves

Transparent shipping tape

Sample containers

Sample/Shipping labels and indelible marker

Bound field log book

PPE

Utility knife

Hip-waders or rubber boots, as necessary

Plastic sheeting

Expanding ruler or tape measure

Variable speed peristaltic bladder or submersible pump with in-line filter or pressurized bailers

Surface water sampling method-specific materials (as necessary):

- Dippers (sampling vessel)
- Pumps with associated tubing
- Double check valve bailers
- new 0.45 micron filter

Decontamination supplies

Procedure:

1. Collection of surface water samples should be completed before collection of the stream sediment samples from the same location. This procedure will eliminate the introduction of sediment and turbulence in the surface water that is to be sampled. If possible, find a suitable sampling location by selecting an area that is away from any sources of cross-contamination that could compromise the integrity of the samples. This includes positioning the sample collection area away from fuel-powered equipment, such as drill rigs or excavators, and upwind of other site activities (e.g., purging, sampling, decontamination) that could influence the sample.

2. Once you have arrived on site and are prepared to conduct the surface water sampling, note all observations and measurements in the field book.
3. Perform a quick reconnaissance of the site to identify sampling locations.
4. Record the approximate ambient air temperature, precipitation, wind (direction and speed), tidal, and other field conditions in the field book. In addition, any site-specific conditions or situations that could potentially affect the sediment samples or associated water level measurements at the sample locations should be recorded.
5. Describe the sampling location including width of stream, depth of stream, water color and clarity (transparency), and approximate surface flow (e.g., slow, fast moving, etc.)
6. Label all sample containers prior to sample collection.
7. The sampler should wear hip-waders or rubber boots and gloves, or Saranex or Tyvek sleeves duct taped onto nitrile gloves, to avoid dermal contact with the surface water. Wear a clean pair of new, disposable gloves each time a different sample is collected and don the gloves immediately prior to collection to limit the possibility of cross-contamination from accidental contact.
8. Collect all samples using either new, disposable equipment, or properly decontaminated sampling equipment. The equipment should be constructed of non-reactive, non-leachable materials (e.g., stainless steel, Teflon®, Teflon®-coated steel, polyethylene, polypropylene, etc.) which are compatible with the chemical constituents at the site.
9. Avoid wading into surface water, if possible, to avoid sediment suspension and potential cross-contamination. If necessary, extreme caution should be exercised when wading into the stream at the sampling location to minimize disturbance of the fine sediments.
10. Because of possible unseen water hazards, two people should be present during the collection of surface water samples.
11. If collecting several surface water samples from the same surface water body, start sampling at the downstream location and progressively move upstream. The sampler should always face upstream (i.e., upcurrent) when collecting the surface water sample.
12. The surface water sample container should be placed into the flowing water and the sample should be collected from just beneath the stream surface. Surface water should be allowed to slowly enter the sampling vessel until the necessary sample volume has been collected. Position the sampling vessel in the upstream direction of flow to minimize the disturbance to the surface water. For dissolved metals analysis, surface water samples must be filtered using the following procedures:
    - Use a variable speed peristaltic, bladder, or submersible pump with the in-line filter fitted on the outlet end; pressurized bailers can also be used.
    - At the pump discharge end, attach a clean 0.45 micron filter to the tubing.
    - Place the inlet into the sampling vessel, turn on the pump and reduce the flow rate, but maintain a flow rate high enough to deliver a smooth stream of water without splashing or undue agitation, hold the filter upright with the inlet and outlet in the vertical position and pump surface water through the filter until all atmospheric oxygen has been removed and the minimum volume of water has been flushed through the filter.

- Collect the filtered samples directly into the laboratory supplied preserved (nitric acid) sample container from the pump-filter assembly.
- If sediment is visible in the sample container after filtration, filter break-through has occurred and the sampling and filtering process should be repeated.
- Disassemble the pump head and discard the tubing and filter appropriately.

13. The sample container should be labeled before sample collection. After the sample is collected, the outside of the container should be cleaned, dried, and sealed. Complete all required information (sample number, date, time, sampler's initials, analysis, preservatives, place of collection) on the sample label. Place clear tape over the tag or label. Immediately after collection, preserve samples for temperature-sensitive parameters by placing them into an insulated cooler for shipment to the analytical laboratory.
14. The sampling location should be described, including width of stream, depth of stream, water color, and approximate surface flow (e.g., slow, fast moving, etc.).
15. Sampling locations should be marked with a stake or flagged for future reference. Locations should be recorded with respect to a permanent feature, if available.
16. Complete chain of custody form with appropriate sampling information.
17. Discard the used hose and filter and utilize new hose and filters for each sample location.
18. If collecting sediment samples, proceed to collect the sample from this location.

Surface Water Field Measurements

Materials:

Nitrile gloves

Saranex or Tyvek suit

Vinyl gloves

Sample containers

Sample/Shipping labels and indelible marker

Transparent shipping tape

Bound field log book

PPE

Utility knife

Hip-waders or rubber boots, as necessary

Aluminum foil or heavy-duty zipper-style plastic bags (quart size)

Plastic sheeting

Expanding ruler or tape measure

Variable speed peristaltic bladder or submersible pump with in-line filter or pressurized bailers

Surface water sampling method-specific materials (as necessary):

- Dippers (sampling vessel)
- Pumps with associated tubing
- Double check valve bailers
- 0.45 micron filter

Decontamination supplies

Procedure:

1. Collect all samples using either new, disposable equipment, or properly decontaminated sampling equipment. The equipment should be constructed of non-reactive, non-leachable materials (e.g., stainless steel, Teflon®, Teflon®-coated steel, polyethylene, polypropylene, etc.) which are compatible with the chemical constituents at the site.
2. Collect all samples for indicator parameters, including pH, temperature, turbidity, and electrical conductivity in accordance with the project-specific work plan and manufacturer's specifications for field testing equipment.
3. Record the date and time of the measurement, temperature, field/weather conditions, surface water level measurements, and visual observations including color, flow direction, and approximate gradient.
4. Field samples must be directly transferred from the sampling equipment to the container that has been specifically prepared for that given parameter; intermediate containers should be avoided.
5. Field instruments will be calibrated prior to field work in accordance with manufacturer instructions and cleaned in the field between samples to prevent cross contamination.
6. Temperature, pH, and electrical conductivity will be measured with a Hach 2100Q turbidimeter or equivalent and turbidity will be measured with a Myron L Ultrameter II meter or equivalent
7. Prior to sampling in the field, pH meters will be calibrated with buffer solutions to manufacturer specification as indicated in its operations and maintenance manual.
8. Temperature measurements will be measured with thermometers calibrated using a National Institute of Standards and Technology (NIST) traceable thermometer. NIST traceable thermometers are certified by the manufacturer as indicated in its operations and maintenance manual.
9. Turbidity will be measured with a nephelometric turbidity units (NTU) range turbidimeter calibrated daily to a reference standard in accordance to the manufacturer's operation and maintenance manual. Sample and standards cuvets must be handled with care as fingerprint smudges, scratches, condensation, and agitation may cause measurement errors. Outside of cuvettes should be cleaned to remove any smudges or condensation.
10. If possible, avoid floating debris, coarse sediment, and fine air bubbles when collecting turbidity samples as these may cause inaccurate readings.

Sediment Sampling

Materials:

Hand trowels, shovels, or spoons (stainless steel or Teflon)
Nitrile or vinyl gloves

Saranex or Tyvek suit
Hip-waders or rubber boots, as necessary
Sample containers
Sample/Shipping labels and indelible marker
Transparent shipping tape
Bound field log book
PPE
Utility knife
Mixing tray or bowl
Heavy-duty zipper-style plastic bags (quart or snack size)
Plastic sheeting
Expanding ruler or tape measure
Munsell color chart
Field test kits, as needed
Sediment sampling method specific materials:
Stainless steel trowels, shovels, or spoons
Bucket augers, auger extension rods, auger handle, pipe wrenches
Split-spoon samplers, pipe wrenches
Direct push acetate liners
Shelby tube samplers
Decontamination supplies

Procedure:

1. Once you have arrived on site and are prepared to conduct the sediment sampling, note all observations and measurements in the field book.
2. Perform a quick reconnaissance of the site to identify sampling locations.

3. Record the approximate ambient air temperature, precipitation, wind (direction and speed), tidal, and other field conditions in the field book. In addition, any site-specific conditions or situations that could potentially affect the sediment samples or associated water level measurements at the sample locations should be recorded.
4. Describe the sampling location including width of stream, depth of stream, water color and clarity (transparency), and approximate surface flow (e.g., slow, fast moving, etc.)
5. Mark sampling locations with a stake or flag for future reference; if available, record locations with respect to a permanent feature.
6. Label all sample containers prior to sample collection.
7. A hand trowel can be used to sample shallow stream bottom sediments, where the depth of water does not exceed 1 foot, using the same procedures specified in the Standard Operating Procedures for Collection of Soils Samples Using a Hand Trowel. A sediment corer or other appropriate sampling device should be used in water deeper than 1 foot (see SOP-6).
8. The sampler should wear hip-waders or rubber boots and gloves, or Saranex or Tyvek sleeves duct taped onto nitrile gloves, to avoid dermal contact with the water.
9. Extreme caution should be exercised when wading into the stream at the sampling location to minimize disturbance of the fine sediments.
10. If collecting several sediment samples from the stream, start sampling at the downstream location and progressively move upstream. The sampler should always face upstream (into the current) when collecting the sediment sample.
11. Position fuel powered equipment downwind and at least 10 feet from the sampling location; make sure that the exhaust faces downwind.
12. Insert the trowel into the sediment bed and retrieve sediment. Carefully remove the trowel from the water to avoid washing sediment from the blade. The trowel blade should be tilted at a slight angle, if necessary, to drain excess water from the blade before placing the sediment in the mixing tray. Separate clumps of sediment material and mix the sediments to a homogeneous particle size and texture. Transfer the contents to the appropriate sample container using a stainless steel spoon.
13. If more sediment is needed to provide sufficient sample volume, reinsert trowel at the sample location and retrieve as before.
14. After the sample is collected, clean the outside of the sample container to remove excess sediment and seal the container. Record all required information (sample number, date, time, sampler's initials, analysis, preservatives, place of collection) on the sample label. Place clear tape over the tag or label. Immediately after collection, preserve samples for temperature-sensitive parameters by placing them into an insulated cooler maintained at a temperature of approximately 4°Celcius.
15. Prepare the sediment samples for offsite laboratory analysis. Record sample designation, date, time, and the sampler's initials on the sample tracking form and in the field book.
16. Complete chain-of-custody forms with appropriate sampling information.
17. Complete sample packing and shipping in accordance with proper procedures.

Sediment Field Measurements

Materials:

PPE

Hip-waders or rubber boots, as necessary

Hand trowels, shovels, or spoons (stainless steel or Teflon)

pH meter (Horiba D51 or equivalent)

Sample containers

Sample/Shipping labels and indelible marker

Bound field log book

Utility knife

Mixing tray or bowl

Heavy-duty zipper-style plastic bags (quart or snack size)

Plastic sheeting

Expanding ruler or tape measure

Munsell color chart

Field test kits, as needed

Sediment sampling method specific materials:

Stainless steel trowels, shovels, or spoons

Decontamination supplies

Procedure:

1. Collect all samples using either new, disposable equipment, or properly decontaminated sampling equipment. The equipment should be constructed of non-reactive, non-leachable materials (e.g., stainless steel, Teflon®, Teflon®-coated steel, polyethylene, polypropylene, etc.) which are compatible with the chemical constituents at the site.
2. Collect all samples for indicator parameters, including pH in accordance with the project-specific work plan and manufacturer's specifications for field testing equipment.
3. Field instruments will be calibrated in accordance with manufacturer instructions and cleaned in the field between samples to prevent cross contamination.
4. Record the date and time of the measurement, temperature, field/weather conditions, and visual observations including color, texture, odor, presence of oil sheens, and presence of macrophites or animals.
5. Field samples must be directly transferred from the sampling equipment to the container that has been specifically prepared for that given parameter; intermediate containers should be avoided.
6. Field instruments will be calibrated in accordance with manufacturer instructions and cleaned in the field between samples to prevent cross contamination.

7. pH will be measured with a Horiba D51 or equivalent that has been calibrated to manufacturer's specifications as indicated in its operations and maintenance manual.
8. Prior to sampling in the field, pH meters will be calibrated with buffer solutions to manufacturer specification as indicated in its operations and maintenance manual.
9. Sediment classification should be performed whenever samples are being collected to provide context for the analysis. Unified Soil Classification System (USCS) logging procedures as described in ATSM D2488[1] are preferred . The emphasis of sediment classification in the field must be on describing the sediments using ALL of the required descriptors; categorization of the USCS group name or symbol alone may not provide details about the soils that could later prove useful. Avoid geologic interpretation or the use of local formation names, which are often difficult to determine in the field without the regional framework. Record ALL of the following information for each soil type:
   - Depth interval
   - USCS group name
   - USCS group symbol
   - Color, using Munsell chart (in moist condition)
   - Percent of cobbles or boulders, or both (approximate; by volume)
   - Percent of gravel, sand, or fines, or all three (approximate; by dry weight)
   - Particle-size range:
     - Gravel—fine, medium, coarse
     - Sand—fine, medium, coarse
   - Particle angularity: angular, subangular, subrounded, rounded
   - Particle shape: (if appropriate) flat, elongated, flat and elongated
   - Maximum particle size or dimension
   - Hardness of coarse sand and larger particles
   - Plasticity of fines: non-plastic, low, medium, high
   - Dry strength: none, low, medium, high, very high (does not apply to sands)
   - Dilatancy: none, slow, rapid
   - Toughness: low, medium, high
   - Odor (mention only if organic or unusual)
   - Moisture: dry, moist, wet
   - Use the following standard descriptors for the textural percentages:
   - Trace: 0 to 10%
   - Little: 11 to 20%
   - Some: 21 to 35%
   - And: 36 to 50%

Example descriptions, using the information listed above, would read as follows:
8-10' – 5YR2/6 fine- to medium-grained sand, trace medium sub-angular rounded gravel (up to 0.5" in diameter); medium dense to dense; wet with slow dilatancy; moderate solvent-like odor between 9' and 10'.

10-12' – 5YR2/6 low plasticity clay with some fine to coarse grained angular to subangular gravels (up to 0.25" in diameter) and trace fine to medium grained rounded sands, very stiff, moist with no dilatancy, no odors.

10. Collect field sediment samples by separating clumps of sediment material and mixing the sediments to a homogeneous particle size and texture. Transfer the contents to the appropriate sample container using a stainless steel spoon.

When field indicator parameters require pH measurements for sediment, below is a brief discussion on procedures to follow.

Field Measurements for PH

pH is a measure of the effective concentration (or activity) of hydrogen ions and is expressed as the negative base-10 logarithm of the hydrogen-ion activity in moles per liter. Uncontaminated groundwater typically exhibits a pH ranging from 5 to 9 Standard Units (SU). Changes in pH from background may indicate the presence of groundwater contamination or that existing contamination has spread and can be very useful in identifying well construction or maintenance problems.

After calibrating the monitoring equipment, follow the manufacturer's instructions to select the display for readings the necessary geochemical parameters. The Horiba D51 equipped with a Horiba 9615-10D or 9625-10D pH electrode should be able to penetrate soft sediments. However, penetration into clays, sands and gravel conditions may not be achievable.

The following procedures should be followed and all observations and measurements recorded in the field book.

- Collection of field parameters should be completed within 15 minutes of sample collection
- Immerse the probe in the sediment (in-situ or within the sample container) and wait for stabilization of the reading before recording the measurement.
- If collecting field measurements ex-situ, agitate or swirl the sample during the stabilization period.

Record all field-testing measurement data, to include the following:

- Project name and location
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units

- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

[1] Note that certain states/regulatory programs may require soil classification under a secondary system (e.g., US Department of Agriculture) or the use of hydrochloric acid to test the reaction with soil (none, weak, strong).

Standard Operating Procedure - 5

Sediment/Sludge Sampling with Ekman or Ponar Dredge

Application:

Sediment/sludge samples can be collected with an Ekman dredge, which is normally used when collecting samples from lagoons or other aqueous systems consisting of sediment of a soft consistency such as muck, me, or fine peat. The ponar type dredge is normally used when collecting samples from lagoons or creeks where the sediment is a hard constancy such as sands, gravels or clays. The dredge consists of a spring-loaded set of jaws with a brass sampling bucket that is lowered to retrieve samples. The dredge can be mounted on a pole or suspended from a line. This may not be used for samples to be analyzed for metals and organics.

Materials:

Ekman dredge

Saranex or Tyvek suit

Overboots or hip-waders, as necessary

Stainless steel spoons or scoops

Plastic sheeting or garbage bags

Nitrile, surgical, or work gloves

Trays, mixing pans, or Ziploc® plastic bags

Sample containers

Sample/Shipping labels and indelible marker

Bound field log book

PPE

Utility knife

Heavy-duty zipper-style plastic bags (quart or snack size)

Expanding ruler or tape measure

Munsell color chart

Field test kits, as needed

Decontamination supplies

Procedure:

1. Sampler should don personal protective equipment as required to prevent dermal contact with sample media.
2. Carefully open jaws of the dredge and set trip lines to trigger device.

3. Gently lower the Ekman dredge through the aqueous media until it rests on the sediment.
4. To close the jaws of the dredge, send the messenger down the line; or if the dredge is pole-mounted, strike the spring release button on the top of the pole. The weight will strike the trigger mechanism, causing the spring-loaded jaws to close. This will trap the sludge/sediment inside the sampling bucket.
5. To minimize disturbance and to avoid washing the sample from the dredge bucket, slowly retrieve the dredge through the liquid column above it.
6. The sediment sample should be removed from the dredge bucket with a stainless steel scoop or spoon and transferred directly to the labeled sample container. The sample container should be sealed, labeled, and placed in a cooler with ice or freezer packs for shipment to the analytical laboratory.
7. The sampling locations should be described including width of stream or vessel, depth of stream or vessel, and approximate surface flow rate, if applicable. Locations should be flagged or marked with a stake for future reference.
8. Keep detailed notes in the field log book of the sample location, sample description, sample length, sampler's name, and the requested analytical parameters.
9. Completed chain-of-custody forms with appropriate sampling information.

Sediment Field Measurements

Materials:

Hand trowels, shovels, or spoons (stainless steel or Teflon)

Nitrile gloves

Vinyl gloves

Aluminum foil

Saranex or Tyvek suit

Hip-waders or rubber boots, as necessary

Sample containers

Sample/Shipping labels and indelible marker

Bound field log book

PPE

Utility knife

Mixing tray or bowl

Heavy-duty zipper-style plastic bags (quart or snack size)

Plastic sheeting

Expanding ruler or tape measure

Munsell color chart

Field test kits, as needed

Sediment sampling method specific materials:

Stainless steel trowels, shovels, or spoons

Bucket augers, auger extension rods, auger handle, pipe wrenches

Split-spoon samplers, pipe wrenches

Direct push acetate liners

Shelby tube samplers

Decontamination supplies

Procedure:

11. Collect all samples using either new, disposable equipment, or properly decontaminated sampling equipment. The equipment should be constructed of non-reactive, non-leachable materials (e.g., stainless steel, Teflon®, Teflon®-coated steel, polyethylene, polypropylene, etc.) which are compatible with the chemical constituents at the site.
12. Collect all samples for indicator parameters, including pH in accordance with the project-specific work plan and manufacturer's specifications for field testing equipment.
13. Field instruments will be calibrated in accordance with manufacturer instructions and cleaned in the field between samples to prevent cross contamination.
14. Record the date and time of the measurement, temperature, field/weather conditions, and visual observations including color, texture, odor, presence of oil sheens, and presence of macrophites or animals.
15. Field samples must be directly transferred from the sampling equipment to the container that has been specifically prepared for that given parameter; intermediate containers should be avoided.
16. Field instruments will be calibrated in accordance with manufacturer instructions and cleaned in the field between samples to prevent cross contamination.
17. pH will be measured with a Horiba D51 or equivalent that has been calibrated to manufacturer's specifications as indicated in its operations and maintenance manual.
18. Prior to sampling in the field, pH meters will be calibrated with buffer solutions to manufacturer specification as indicated in its operations and maintenance manual.
19. Sediment classification should be performed whenever samples are being collected to provide context for the analysis. Unified Soil Classification System (USCS) logging procedures as described in ATSM D2488[1] are preferred . The emphasis of sediment classification in the field must be on describing the sediments using ALL of the required descriptors; categorization of the USCS group name or symbol alone may not provide details about the soils that could later prove useful. Avoid geologic interpretation or the use of local formation names, which are often difficult to determine in the field without the regional framework. Record ALL of the following information for each soil type:
    - Depth interval
    - USCS group name
    - USCS group symbol

- Color, using Munsell chart (in moist condition)
- Percent of cobbles or boulders, or both (approximate; by volume)
- Percent of gravel, sand, or fines, or all three (approximate; by dry weight)
- Particle-size range:
  - Gravel—fine, medium, coarse
  - Sand—fine, medium, coarse
- Particle angularity: angular, subangular, subrounded, rounded
- Particle shape: (if appropriate) flat, elongated, flat and elongated
- Maximum particle size or dimension
- Hardness of coarse sand and larger particles
- Plasticity of fines: non-plastic, low, medium, high
- Dry strength: none, low, medium, high, very high
- Dilatancy: none, slow, rapid
- Toughness: low, medium, high
- Odor (mention only if organic or unusual)
- Moisture: dry, moist, wet
- Use the following standard descriptors for the textural percentages:
- Trace: 0 to 10%
- Little: 11 to 20%
- Some: 21 to 35%
- And: 36 to 50%

Example descriptions, using the information listed above, would read as follows:
8-10' – 5YR2/6 fine- to medium-grained sand, trace medium sub-angular rounded gravel (up to 0.5" in diameter); medium dense to dense; wet with slow dilatancy; moderate solvent-like odor between 9' and 10'.

10-12' – 5YR2/6 low plasticity clay with some fine to coarse grained angular to subangular gravels (up to 0.25" in diameter) and trace fine to medium grained rounded sands, very stiff, moist with no dilatancy, no odors.

Collect field sediment samples by separating clumps of sediment material and mixing the sediments to a homogeneous particle size and texture. Transfer the contents to the appropriate sample container using a stainless steel spoon.

When field indicator parameters require pH measurements for sediment, below is a brief discussion on procedures to follow.

Field Measurements for PH

pH is a measure of the effective concentration (or activity) of hydrogen ions and is expressed as the negative base-10 logarithm of the hydrogen-ion activity in moles per liter. Uncontaminated groundwater typically exhibits a pH ranging from 5 to 9 Standard Units (SU). Changes in pH from background may indicate the presence of groundwater contamination or that existing contamination has spread and can be very useful in identifying well construction or maintenance problems.

After calibrating the monitoring equipment, follow the manufacturer's instructions to select the display for readings the necessary geochemical parameters. The Horiba D51 equipped with a Horiba 9615-10D or 9625-10D pH electrode should be able to penetrate soft sediments. However, penetration into clays, sands and gravel conditions may not be achievable.

The following procedures should be followed and all observations and measurements recorded in the field book.

- Collection of field parameters should be completed within 15 minutes of sample collection
- Immerse the probe in the sediment (in-situ or within the sample container) and wait for stabilization of the reading before recording the measurement.
- If collecting field measurements ex-situ, agitate or swirl the sample during the stabilization period.

Record all field-testing measurement data, to include the following:

- Project name and location
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

Standard Operating Procedure - 6

Sediment Sampling Using Hand Corer

Application:

Sampling bottom sediment in shallow surface water bodies and lagoons

Materials:

Hand corer

Extension handle

Analyte-inert core tube liners (glass, Teflon, etc.)

Nitrile, surgical, or work gloves

Protective overboots

Tray, mixing pans, or Ziploc® plastic bags

Stainless steel spoons or trowel

Pipe wrenches

Bound log book

Assembly:

1. Secure the core tube head assembly onto the core tube, as specified by manufacturers' instructions.
2. Remove end liner caps and wrapping from a clean core tube liner and insert the liner into the core tube.
3. Insert a clean core retainer into the bottom of the core tube liner so that its convex end points away from the bottom. Plastic materials should not be used if organic analytes are of concern.
4. Screw the shoe (nose cone) onto the bottom of the core tube. The hand corer is now fully assembled for use.
5. If the hand corer is used from a boat or overhead structure, either of the following two attachments can be used:
   a) 5-foot extension handles can be attached to the top of the core head assembly with a threaded adapter fitting, nut, and bolt. The long handle is used to push the corer into bottom sediment.
   b) The corer can be lowered to the bottom with a rope. The rope can be tied to a clevis pin that mounts on the core head assembly with a nut and bolt.

Use:

1. Place nitrile gloves on hands before collecting samples.

2. Sampler should avoid wading into surface water, if possible. If not possible, the sampler should carefully approach the sample location to minimize disturbance of fine sediments.
3. Place the corer in a vertical position perpendicular to the bottom at the desired sample location.
4. The hand corer should be lowered slowly to minimize disturbance of sediments.
5. If using the corer from a wading position push it down into the sediment while turning the handle. The push and twist technique can also be used with the long extension handle attached to the corer.
6. If the corer is attached to a rope, detachable weights can be put on top of the core head assembly by threading the rope through the holes in the center of the weights. The shoe of the corer should not be resting on the bottom before being advanced. Further penetration will be achieved by allowing the corer to fall freely through the water while in a vertical position to strike the bottom. The weights will increase the force of the corer as it strikes the bottom.
7. When sufficient sample has been collected, it is usually necessary to push it back and forth from different angles to loosen the seal between the bottom and the core tube before pulling it up.
8. Slowly retrieve the core tube and place it on a clean surface in a vertical position. Slowly tilt it toward a horizontal position (this can be done immediately following retrieval of the sampling device). This will open the flapper valve and allow water that entered the tube to leak out.
9. Unscrew and remove the core head assembly and shoe from the core tube. Pipe wrenches may be used to unscrew the core tube assembly. Slide the core tube liner out of the core tube.
10. If undisturbed sediment core samples are needed, do not remove the sediment sample from the core tube. Retain the core sample in the core tube liner (i.e., inner sleeve) and place Teflon end caps on each liner end. Label the inner tube and tape the end caps on the tube liner for shipment to the laboratory. Plastic materials, including end cap, should not be used if organic analytes are of concern.
11. To remove sediment from the core tube, place the core tube liner over a sampling tray and remove its contents by inserting the brass rod into the top end of the tube and pushing. The brass rod should be decontaminated and wrapped in clean aluminum foil between each use.
12. If additional sediment is needed to provide necessary sample volume, reassemble the corer using a separate, clean core tube liner and repeat steps 1-11.
13. For volatile organic compound (VOC) samples, do not mix the sample before sampling for VOCs. Transfer sediment from the tray directly into sample containers.
14. Examine the contents of the tray. After the collection of VOC samples and before filling other sample containers, mix the sediment so a homogeneous particle size and texture remain.
15. Using the stainless steel spoon, transfer the tray contents to the sample containers.
16. The sample container should be labeled before sample collection. After the sample is collected, the container should be sealed, wiped clean of excess sediment, and placed in a cooler with ice or freezer packs for shipment to the analytical laboratory.
17. Complete chain-of-custody form with appropriate sample information.
18. The sample location should be described, including depth of the surface water body and approximate surface flow. The location should be flagged or marked for future reference, if possible.

Sediment Field Measurements

Materials:

Hand trowels, shovels, or spoons (stainless steel or Teflon)

Nitrile gloves

Vinyl gloves

Aluminum foil

Saranex or Tyvek suit

Hip-waders or rubber boots, as necessary

Sample containers

Sample/Shipping labels and indelible marker

Bound field log book

PPE

Utility knife

Mixing tray or bowl

Heavy-duty zipper-style plastic bags (quart or snack size)

Plastic sheeting

Expanding ruler or tape measure

Munsell color chart

Field test kits, as needed

Sediment sampling method specific materials:

Stainless steel trowels, shovels, or spoons

Bucket augers, auger extension rods, auger handle, pipe wrenches

Split-spoon samplers, pipe wrenches

Direct push acetate liners

Shelby tube samplers

Decontamination supplies

Procedure:

20. Collect all samples using either new, disposable equipment, or properly decontaminated sampling equipment. The equipment should be constructed of non-reactive, non-leachable materials (e.g., stainless steel, Teflon®, Teflon®-coated steel, polyethylene, polypropylene, etc.) which are compatible with the chemical constituents at the site.
21. Collect all samples for indicator parameters, including pH in accordance with the project-specific work plan and manufacturer's specifications for field testing equipment.

22. Field instruments will be calibrated in accordance with manufacturer instructions and cleaned in the field between samples to prevent cross contamination.
23. Record the date and time of the measurement, temperature, field/weather conditions, and visual observations including color, texture, odor, presence of oil sheens, and presence of macrophites or animals.
24. Field samples must be directly transferred from the sampling equipment to the container that has been specifically prepared for that given parameter; intermediate containers should be avoided.
25. Field instruments will be calibrated in accordance with manufacturer instructions and cleaned in the field between samples to prevent cross contamination.
26. pH will be measured with a Horiba D51 or equivalent that has been calibrated to manufacturer's specifications as indicated in its operations and maintenance manual.
27. Prior to sampling in the field, pH meters will be calibrated with buffer solutions to manufacturer specification as indicated in its operations and maintenance manual.
28. Sediment classification should be performed whenever samples are being collected to provide context for the analysis. Unified Soil Classification System (USCS) logging procedures as described in ATSM D2488[1] are preferred . The emphasis of sediment classification in the field must be on describing the sediments using ALL of the required descriptors; categorization of the USCS group name or symbol alone may not provide details about the soils that could later prove useful. Avoid geologic interpretation or the use of local formation names, which are often difficult to determine in the field without the regional framework. Record ALL of the following information for each soil type:
    - Depth interval
    - USCS group name
    - USCS group symbol
    - Color, using Munsell chart (in moist condition)
    - Percent of cobbles or boulders, or both (approximate; by volume)
    - Percent of gravel, sand, or fines, or all three (approximate; by dry weight)
    - Particle-size range:
      - Gravel—fine, medium, coarse
      - Sand—fine, medium, coarse
    - Particle angularity: angular, subangular, subrounded, rounded
    - Particle shape: (if appropriate) flat, elongated, flat and elongated
    - Maximum particle size or dimension
    - Hardness of coarse sand and larger particles
    - Plasticity of fines: non-plastic, low, medium, high
    - Dry strength: none, low, medium, high, very high
    - Dilatancy: none, slow, rapid

- Toughness: low, medium, high
- Odor (mention only if organic or unusual)
- Moisture: dry, moist, wet
- Use the following standard descriptors for the textural percentages:
- Trace: 0 to 10%
- Little: 11 to 20%
- Some: 21 to 35%
- And: 36 to 50%

Example descriptions, using the information listed above, would read as follows:
8-10' – 5YR2/6 fine- to medium-grained sand, trace medium sub-angular rounded gravel (up to 0.5" in diameter); medium dense to dense; wet with slow dilatancy; moderate solvent-like odor between 9' and 10'.

10-12' – 5YR2/6 low plasticity clay with some fine to coarse grained angular to subangular gravels (up to 0.25" in diameter) and trace fine to medium grained rounded sands, very stiff, moist with no dilatancy, no odors.

1. Collect field sediment samples by separating clumps of sediment material and mixing the sediments to a homogeneous particle size and texture. Transfer the contents to the appropriate sample container using a stainless steel spoon.

When field indicator parameters require pH measurements for sediment, below is a brief discussion on procedures to follow.

Field Measurements for PH

pH is a measure of the effective concentration (or activity) of hydrogen ions and is expressed as the negative base-10 logarithm of the hydrogen-ion activity in moles per liter. Uncontaminated groundwater typically exhibits a pH ranging from 5 to 9 Standard Units (SU). Changes in pH from background may indicate the presence of groundwater contamination or that existing contamination has spread and can be very useful in identifying well construction or maintenance problems.

After calibrating the monitoring equipment, follow the manufacturer's instructions to select the display for readings the necessary geochemical parameters. The Horiba D51 equipped with a Horiba 9615-10D or 9625-10D pH electrode should be able to penetrate soft sediments. However, penetration into clays, sands and gravel conditions may not be achievable.

The following procedures should be followed and all observations and measurements recorded in the field book.

- Collection of field parameters should be completed within 15 minutes of sample collection
- Immerse the probe in the sediment (in-situ or within the sample container) and wait for stabilization of the reading before recording the measurement.

- If collecting field measurements ex-situ, agitate or swirl the sample during the stabilization period.

Record all field-testing measurement data, to include the following:

- Project name and location
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

Standard Operating Procedure - 7

Sludge Sampling with Sludge Judge

Application:

The sludge judge is useful for obtaining a core sample of semi-liquid sludge or water and sludge. This instrument is a long, narrow polyethylene tube with a check valve on the bottom. The tube is graduated in 6-inch intervals for easy identification of sampling depth. A sludge judge may not be used for organics.

Materials:

Sludge judge

Saranex or Tyvek suit

Sample containers, labels, and indelible marker

Tray or mixing pans

Aluminum foil

Plastic sheeting or garbage bags

Nitrile gloves

Vinyl gloves

Eye protection

Over boots

Stainless steel scoops, trowels, or spoons

Procedure:

1. Sampling personnel should don the appropriate personal protective equipment based on the type of material being sampled and its historical characteristics.
2. Slowly insert the sludge judge into the material being sampled.
3. When the sludge judge has filled with material, pull back on the sampler to close the check valve and retrieve the sludge sample.
4. If a liquid sample is not required, decant any liquid into a separate container or back into the vessel being sampled. Transfer the remaining sludge sample directly into a labeled sample container by pouring the sludge from the top of the sampling tube. If a liquid sample is required, decant any liquid in the tube directly into sample containers; retrieve the sludge from tube.
5. The sample container should be sealed, labeled, and placed in a cooler with ice or freezer packs for shipment to the analytical laboratory.
6. Keep detailed notes in the field log book of the sample location, sample description, sample depth, sampler's name, and the requested analytical parameters.
7. Complete chain-of-custody forms with appropriate sampling information

Standard Operating Procedure - 8

Sludge Sampling with Coring Tube

Materials:

Field log book
Personal protective equipment (PPE)
Coring tube
Hammer
Plastic tube liners
Hack saw and replacement blades
Mixing tray or bowl
Plastic sheeting
Stainless steel spoon or trowel
Expanding ruler or tape measure

Note: Decontamination is not required for dedicated sampling equipment.

Procedure:

1. Use appropriate PPE as specified in the site-specific health and safety plan.
2. Insert a plastic liner into the coring tube to collect an undisturbed sample. A plastic core catcher may be used to improve sample recovery.
3. The sample is collected by pushing or hammering the coring tube into the material to the desired depth, making sure not to exceed the length of the coring tube. Continuous core samples can be obtained from stable coreholes by re-inserting the coring device into the same core-hole. However, a new plastic liner must be placed in the coring device before each sample is collected.
4. Remove the sampler by pulling and twisting the coring tube until it breaks free from the material. This will result in a core that is approximately 2 feet long.
5. If the sample needs to be collected from a depth greater than 2 feet, place a new plastic liner into the tube and re-insert the sampler into the same core-hole and repeat Steps 3 and 4.
6. Remove the plastic liner from the coring tube and decant any excess water above the core.
7. Cut the plastic liner at the desired sample interval using a hack saw with a decontaminated blade. For VOC samples, extrude the sample material directly into a sample container. A closed-system sampler (e.g., Encore Sampler) should be used, if necessary, to collect sludge samples for VOC analysis using EPA Method 5035 for preservation.

8. For all other parameters, a stainless steel spoon or trowel should be used to transfer the sample material into a decontaminated mixing tray or bowl to be homogenized. The decontaminated stainless steel spoon or trowel can be used to remove gummy or thick sludge from the tube, if required.
9. If necessary, screen the sludge with a PID/OVA or perform a headspace analysis in accordance with SOP 22. Record the reading in the field logbook.
10. Record the sample location, sample description, sample depth, sampler's name, and the requested analytical parameters in the field log book.
11. Label the containers, cover the labels with tape, and immediately place the containers in a cooler maintained at an ambient temperature of 4* Celsius with wet ice. Freezer packs or dry ice should not be used for sample preservation.
12. Complete chain-of-custody forms with appropriate sampling information.
13. Samples should be maintained and shipped in accordance with SOP 20.
14. Properly manage all PPE and investigation-derived wastes in accordance with state and federal requirements.

Standard Operating Procedure – 9

Soil Sampling Using Bucket Auger

Materials:

Field log book

Personal protective equipment (PPE)

Bucket augers

Auger extension rods

Auger handle

Pipe wrenches (for threaded connections)

Push pins (for snap connections)

Stainless steel spoons or trowels

Mixing tray or bowl

Plastic sheeting

Expanding ruler or tape measure

Note: Decontamination is not required for dedicated sampling equipment.

Procedure:

1. Use appropriate PPE as specified in the site-specific health and safety plan.
2. Remove all vegetation or other surface material (e.g., gravel) with a hand trowel or other tool (e.g., shovel).
3. Advance the borehole to the desired sampling depth (i.e., the top of the sample interval). Attach a decontaminated auger bucket to collect the soil sample.
4. Place the auger bucket in the borehole. Grip the cross-handle with both hands and twist it clockwise to advance the auger.
5. Withdraw the auger bucket from the borehole and place it on plastic sheeting. For VOC samples, use a decontaminated stainless steel spoon or trowel to transfer the sample material directly into the appropriate sample container. A closed-system sampler (e.g., Encore Sampler) should be used, if necessary, to collect sludge samples for VOC analysis using EPA Method 5035 for preservation.
6. Remove the retrieved soil from the bucket with a decontaminated stainless steel spoon or trowel and place the material in a decontaminated mixing tray or bowl. If additional soil is needed to provide sufficient sample volume, repeat Step 4.
7. If necessary, screen the lead end of the auger with a PID/OVA or perform headspace analysis in accordance with SOP 22. Record the reading in the field logbook.

8. Describe the remaining sample material in accordance with ASTM International Standard D 2488 and the Unified Soil Classification System. Record the sample description in the field logbook.
9. For all other parameters, the sample material should be transferred into a decontaminated mixing tray or bowl. Use the stainless steel spoon to separate large clumps of soil material and mix the contents of the tray to a homogeneous particle size and texture.
10. Examine the contents of the tray and remove coarse gravel, organic material (e.g., roots, grass, and woody material) and any other debris with the stainless steel spoon.
11. Transfer the tray contents to the appropriate sample container using a stainless steel spoon.
12. Label the containers, cover the labels with tape, and immediately place the containers in a cooler maintained at an ambient temperature of 4* Celsius with wet ice. Freezer packs or dry ice should not be used for sample preservation.
13. Measure and record the sample depth in the field logbook, along with the sample location, sampler name, and the requested analytical parameters.
14. Complete the chain-of-custody form with appropriate sampling information.
15. Samples should be maintained and shipped in accordance with SOP 20.
16. Properly manage all PPE and investigation-derived wastes in accordance with state and federal requirements.

Standard Operating Procedure - 10

Split-Spoon Soil Sampling

Application:

To collect soil samples with a split-spoon sampler that is advanced and retrieved with a drill rig.

Materials:

Field log book

Personal protective equipment (PPE)

Split-spoon samplers

Stainless steel spoons or trowels

Mixing tray or bowl

Pipe wrenches

Expanding ruler or tape measure

Note: Decontamination is not required for dedicated sampling equipment.

Procedure:

1. Use appropriate PPE as specified in the site-specific health and safety plan.
2. Ensure that the soil boring has reached the desired sample depth and that loose soil in the bottom of the boring has been cleaned out.
3. The drillers will attach a decontaminated split-spoon sampler to the lead drilling rod, lower it to the bottom of the boring, and advance the split-spoon into the undisturbed soil. Record in the field logbook the number of blows for every 6 inches the sampler is advanced.
4. When the split-spoon has reached the desired depth, the driller will retrieve the split-spoon and disconnect the split-spoon from the drilling rod.
5. Remove the head and shoe from the split-spoon and note which end of the sampler is the top and bottom. Separate the split-spoon sampler into two halves keeping all of the soil in one of the halves (if possible).
6. Measure the length of material recovered in the sampler with respect to the penetration depth and record this ratio in the field logbook.
7. If field screening for organic vapors is required, break or cut the soil core every 3 to 4 inches and quickly scan the breaks in the core material with a PID/OVA. Headspace analyses, if required, should be performed in accordance with SOP 22. Record the reading in the field logbook.
8. For VOC samples, transfer the soil directly into the sample containers with a decontaminated stainless steel spoon. A closed-system sampler (e.g., Encore Sampler) should be used, if necessary, to collect sludge samples for VOC analysis using EPA Method 5035 for preservation.

9. Describe the remaining sample material in accordance with ASTM International Standard D 2488 and the Unified Soil Classification System. Record the information in the field logbook.
10. Note: the top of the split-spoon often contains float material, which is not to be included in the soil sample or sample description.
11. If it is necessary to collect soil samples for non-volatile parameters, transfer the recovered soil to a decontaminated mixing tray or bowl with a decontaminated stainless steel spoon. Soil material lodged within the split-spoon shoe should also be placed in the mixing tray or bowl for sampling.
12. Examine contents of the tray and remove pebbles, organic material, (e.g., roots, grass, and woody material), and other debris with the stainless steel spoon. Use the same spoon to chop apart clumps of soil and mix the contents of the tray to a homogeneous particle size and texture.
13. Transfer the soil material to the appropriate sample containers using the stainless steel spoon.
14. Label the containers, cover the labels with tape, and immediately place the containers in a cooler maintained at an ambient temperature of 4* Celsius with wet ice. Freezer packs or dry ice should not be used for sample preservation.
15. Record the sample location, sample depth, sampler name, and the requested analytical parameters in the field log book.
16. Complete the chain-of-custody form with appropriate sampling information.
17. Samples should be maintained and shipped in accordance with SOP 20.
18. Properly manage all PPE and investigation-derived wastes in accordance with state and federal requirements.

BORING LOG WSP Environment & Energy 11190 Sunrise Valley Drive, Suite 300 Reston, Virginia 20191	**PROJECT**	Boring No. Sheet ___ of Date Drilled

Drilling Co. Driller Geologist	Boring Location Ground Elevation TOC Elevation

Boring	Casing/S	Sampler
Method Hole Diameter Inside Diameter Total Depth	Type Diameter Screen Length Screen Slot Size	Method Length (ft) Hammer (lb) Fall (in)

Depth	P.I.D. (ppm)	Percent Recovery	Sample Depth	Blows/6"	Well Design	Sample Description

Standard Operating Procedure - 11

Soil Sampling Using Hand Trowel

Application:

To collect surface or shallow subsurface soil samples with a hand trowel.

Materials:

Field log book

Personal protective equipment (PPE)

Stainless steel trowels or spoons

Mixing tray or bowl

Plastic sheeting

Note: Decontamination is not required for dedicated (disposable) sampling equipment.

Procedure:

1. Use appropriate PPE as specified in the site-specific health and safety plan.
2. Remove any vegetation or other surface material (e.g., gravel) from the sample location with a trowel or other tool (e.g., shovel).
3. Push the trowel into the soil to the desired sampling depth. If sampling a stiff silty or clayey soil, it may be necessary to remove and reinsert the trowel to loosen the soil. Shallow subsurface soil samples can be collected by digging a hole (e.g., with a shovel or trowel) and collecting a soil sample at the desired depth.
4. Transfer the soil to a mixing tray or bowl using a decontaminated stainless steel trowel or spoon. If required, screen the recovered soil with a PID/OVA or perform headspace analyses in accordance with SOP 22. Record the reading in the field logbook.
5. For VOC samples, transfer soil directly into the sample container with the stainless steel trowel. A closed-system sampler (e.g., Encore Sampler) should be used, if necessary, to collect soil samples for VOC analysis using EPA Method 5035 for preservation.
6. Describe the remaining sample material in accordance with ASTM International Standard D 2488 and the Unified Soil Classification System. Record the sample description in the field logbook.
7. If soil samples will be collected for non-volatile parameters, use the stainless steel spoon to chop apart clumps of soil material and mix the contents of the tray to a homogeneous particle size and texture.
8. Examine contents of the tray and remove pebbles, organic material, (e.g., roots, grass, and woody material), and other debris with a stainless steel trowel or spoon.
9. Transfer the tray contents to the appropriate sample container using a stainless steel spoon.

10. Label the containers, cover the labels with tape, and immediately place the containers in a cooler maintained at an ambient temperature of 4* Celsius with wet ice. Freezer packs or dry ice should not be used for sample preservation.
11. Record the sample location, sample depth, sampler name, and the requested analytical parameters in the field log book.
12. Complete the chain-of-custody form with appropriate sampling information.
13. Samples should be maintained and shipped in accordance with SOP 20.
14. Properly manage all PPE and investigation-derived wastes in accordance with state and federal requirements.

Standard Operating Procedure - 12

Chip, Wipe, and Sweep Sampling

Application:

These methods of sample collection are intended for monitoring surficial contamination of nonvolatile species of analytes on porous surfaces for chip samples and smooth surfaces for wipe samples. Also included is a method for collecting dust samples from porous or non-porous surfaces.

Materials:

Lab-clean sample containers of proper size and composition

Field and travel blanks

Site log book

Sample analysis request forms

Chain of custody forms

Custody seals

Sample seals

Disposable surgical gloves

Sterile wrapped gauze pad (3 in. X 3 in.)

Appropriate pesticide (HPLC) grade solvent

Medium-sized, laboratory-cleaned paint brush

Medium-sized, laboratory-cleaned chisel

Autoclaved aluminum foil

Camera

Distilled/deionized water

Procedure:

1.1.8 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed.
2. Obtain necessary sampling and monitoring equipment. Decontaminate or pre-clean the equipment, and ensure that it is in working order.
3. Perform a general site survey and mark all sampling locations. Measure off the designated area and photo document.

1.1.9 Chip Sample Collection

1. Don a new pair of disposable surgical gloves. Remove aluminum foil wrap from the laboratory cleaned chisel or equivalent sampling device.
2. Chip the sample area horizontally, then vertically to an even depth of approximately 1/8 inch. Collect the chipped fragments using a decontaminated dustpan and bristle brush and transfer the sample directly into the labeled sample bottle. Cap the container, attach the label and custody seal, and place in a double plastic bag. Store the samples out of direct sunlight and cool to 40 Celsius.
3. Record all pertinent data (sample location, sample description, sample depth, sampling personnel, and the requested analytical parameters) in the site logbook. Complete chain-of-custody forms with appropriate sampling information.

1.1.10 Wipe Sample Collection

1. Don a new pair of disposable surgical gloves. Open a new sterile package of gauze pad. Soak the pad with the appropriate solvent (one in which the contaminant is most soluble). Wipe the marked surface area using firm strokes. Wipe vertically, then horizontally to ensure complete surface coverage.
2. Place the gauze pad in an appropriately prepared sample container with a Teflon-lined cap. Cap the sample container, attach the label and custody seal, and place in a double plastic bag.
3. Record all pertinent data (sample location, sample description, sample depth, sampling personnel, and the requested analytical parameters) in the site logbook. Complete the chain-of-custody forms before taking the next sample.
4. A blank sample should be collected for each sampling event. This consists of a sterile gauze pad, wet with the appropriate solvent, and placed in a prepared sample container. The blank will help identify potential introduction of contaminants via the sampling methods, the pad, solvent, or sample container.
5. Store samples out of direct sunlight and cool to 40 Celsius.

1.1.11 Sweep Sample Collection

1. Don a new pair of disposable surgical gloves. Sweep the measured area using a dedicated brush and collect the sample in a dedicated dustpan (pre-cleaned).
2. Transfer the sample from the dustpan to the sample container. Cap the sample container, attach the label and custody seal, and place in a double plastic bag.
3. Record all pertinent data (sample location, sample description, sample depth, sampling personnel, and the requested analytical parameters) in the site logbook. Complete the chain-of-custody form before taking the next sample.
4. Store samples out of direct sunlight and cool to 40 Celsius.

Standard Operating Procedure - 13

Concrete Core Sampling

Materials:

Concrete corer and drill
Tap water or containers of water
Concrete chisel
Hammer
Aluminum foil
Ionic detergent
Wet/dry vacuum
Stiff-bristled brushes
Sandpaper
Nitrile gloves
Respiratory protection
Tyvek suits
Paper towels
Eye protection
Indelible marker
Kimwipes
Generator (i.e., electrical power)
Extension cord (heavy duty)
Pipe wrenches
Work gloves
Garden hose

Procedure:

1. Clean surface area to be sampled by scrubbing it using ionic detergent (e.g., Whisk solution) and stiff-bristled brush. Rinse the surface with deionized water, then remove the water by vacuuming surface with wet/dry vacuum.
2. Remove any sealant coatings from the surface area using sandpaper or a grinder. Wash, rinse, and vacuum the surface area.
3. Move the drill into position and connect the decontaminated concrete corer to the drill. Connect the water supply and pressure distribution tank.

4. Establish vacuum pressure to hold core drilling rig to the floor.
5. Lower the corer to within 2 inches of the surface, start the drill, and slowly lower the corer to the surface.
6. Keep a steady stream of water flowing through the corer to cool the concrete, prevent the concrete core from splitting, and control fugitive dust. Do not remove corer from the hole until the required depth is reached. However, should the concrete corer become lodged, shut off power immediately to avoid injury.
7. With the drill still operating, remove the corer from core hole and shut off. Observe the hole to see if core was removed. If the concrete core is still in the floor or the core barrel, use a concrete chisel and rubber mallet to break the core loose.
8. When removing the core from the corer, handle the core with clean nitrile gloves.
9. Place a label on the top of the core written with a permanent marking pen. DO NOT WRITE on the core itself, especially if volatile organic compound analysis is requested. Measure the total length of core and record it in the field log book. Wrap the core in aluminum foil and place it in a labeled sample container.
10. Keep detailed notes in the field log book of the sample location, sample description, sample depth, sampling personnel, and the requested analytical parameters.
11. Complete chain-of-custody forms with appropriate sampling information

Standard Operating Procedure - 14

Waste Pile Sampling

Application:

As with soil sampling, waste pile samples can be collected at the surface or at depth, and different equipment is required in each instance. Surface samples can be collected most efficiently with a trowel or scoop. For samples at depth, a decontaminated, clean bucket auger may be required. For a sample core, waste pile samplers may be used. To collect representative samples of a waste pile, composite samples may be required. The objectives of the sampling program will dictate the need to collect composite samples. A grid system set-up on the waste pile may also be used to collect individual or composite samples.

Materials:

- Stainless steel trowels or scoops
- Stainless steel spoons
- Bucket augers
- Extension rods
- Auger handle
- Pipe wrenches
- Teflon tape
- Work, nitrile, and surgical gloves
- Saranex or Tyvek suit
- Eye protection
- Respirator (if necessary)
- Analytical buckets
- Tray, mixing pans
- Ziploc® plastic bags
- Aluminum foil
- Stakes and string (grid system)
- Plastic sheeting
- HNu or other photoionization detector (PID)

Procedure:

1. At the desired sampling location, clear away the accumulated surface debris.

2. The sampler should put on the correct personal protective equipment as dictated by the medium to be sampled and the project. Care should be taken to ensure that contact between the waste material and the skin, eyes, and face of the sampling personnel is prevented.
3. If a grid system is being employed for sampling the waste pile, lay out the grid according to the developed sampling plan.
4. Assemble the sample equipment (e.g., bucket augers) if necessary, and collect the samples at the chosen designations.
5. Collect an adequate volume of waste from a depth of 0-6 inches using the bucket auger, trowel, or scoop at the desired sampling locations. Record the PID measurement in the open borehole.
6. If using a bucket auger, advance the decontaminated bucket auger by turning the bucket auger clockwise into the sample medium. To remove the auger, turn the auger counter-clockwise and pull it from the borehole. This procedure is repeated until sampling is complete. If the sampled waste pile is heterogeneous, a clean, decontaminated bucket must be used every time to collect the sample of the waste material, if necessitated by the objectives of the sampling plan.
7. If composite samples are to be collected, transfer the waste material to a clean tray or mixing pan for compositing. Report the PID measurement of the material in the tray. Use a clean, decontaminated spoon, scoop, or trowel to homogenize the sample. Remove any foreign matter (e.g., rocks, sticks, or other debris) from the tray. Transfer the waste sample directly into a prelabeled sample container. A wide-mouth sampling container is preferable for containing the sample, as it requires less disturbance of the waste sample as the sample is being transferred into the labeled sample container.
8. Keep detailed notes in the field log book of the sample location, details about the grid spacing, sample description, sample depth, PID measurements, sampler's name, and the requested analytical parameters.
9. Complete the chain-of-custody forms with the appropriate sampling information..

Standard Operating Procedure - 15

Decontamination of Drilling Equipment

Materials:

Canvas or plastic tarp(s)

4-mil polyethylene liner

Pressurized steam cleaner (steam jenny)

55-gallon steel drums with bung (closed) tops

55-gallon steel drums with open tops, rings, lids, ring-nut and ring-bolt

Hammer, nails, duct tape, extension cord(s)

Wood boards - 4" x 4", 2" x 4" or 2" x6"

Portable wet/dry vacuum

Shovel, funnel, and squeegee

Construction of Decontamination Basin:

1. Place tarp(s) on flat, firm surface in an accessible area of the site away from areas of surface contamination. Use enough tarp to accommodate the rear of the drilling rig and hollow stem augers and to prevent overspray from the steam jenny from falling onto adjacent soil surfaces. If necessary, place more than one tarp on the ground. Overlap tarp edges and secure with duct tape. Area should be slightly inclined toward one corner so that the decontamination water will pool in one corner for easier pumping to the containment drums.
2. Place a layer of polyethylene liner on top of the tarp(s). If one sheet cannot completely cover the tarp, use another one. Overlap the sheets at the edges and secure with duct tape.
3. Place 4" x 4" boards along the tarp's outer edges to form a square or rectangular basin. Roll each 4" x 4" board toward the center so the tarp and polyethylene wrap completely around it at least once. Secure the tarp and liner to the top of the boards with nails, tacks or heavy-duty staples.
4. Place the drums, steam cleaner, and wet/dry vacuum adjacent to one side of the basin on the outside.

Decontamination Procedure:

1. Unload drilling equipment from the drilling rig and place in one side of the basin.
2. Activate the steam cleaner. Personnel performing steam cleaning should don rubber boots, Tyvek or Saranex suits, rubber gloves, and a hard hat with a face shield for splash protection.
3. Clean each piece of drilling equipment, including auger bits, drill bits, portable power augers, hollow stem augers, auger holders, split spoons, rod lifters, and drilling rods, by holding the nozzle of the steam cleaner a few inches away. Wood 2" x 4"s can be placed on the basin floor to

prevent drilling equipment from coming into contact with solids that will build up beneath it as it is being steam cleaned.

4. After each piece is cleaned, place it on rows of 2" x 4" boards in a separate area of the basin.
5. If space allows, position the rear of the drill rig in the basin and use the steam cleaner to clean off rig surfaces and the hoist and derrick as needed.
6. Reload drilling equipment onto rig and drive it out of the basin.
7. Vacuum up liquids on the basin floor with the flexible hose of the portable wet/dry vacuum. A long-handled squeegee can be used to pool liquid together to aid vacuuming.
8. Remove accumulated solids from the basin floor with a shovel and place in open-top drums. During removal of the accumulated solids, be careful so that the polyethylene liner is not torn, cut, or punctured with the shovel.
9. Empty the canister of the wet/dry vacuum into a bung-top drum using a funnel.
10. Secure and tighten tops of drums and apply appropriate hazardous waste or nonhazardous waste labels to each drum. The accumulation date should be placed on each drum. An inventory of all onsite drums should be entered into the field log book by field personnel. All drums should be marked, numbered, or labeled with an indelible marker for future reference.
11. On completion of onsite work, the properly labeled and inventoried drums should be stored within a newly constructed pad or basin until disposal is arranged. This containment area should be constructed of wooden boards with a polyethylene liner, as described above.
12. Materials used in construction of the decontamination basin or pad should be disassembled and placed into a properly labeled drum for future disposal.
13. All drilling equipment and the drill rig should be decontaminated on arrival onsite and before the start of any drilling activity. On completion of site work, the drilling equipment and rig should be decontaminated by the drilling contractor before departure from the site.

Standard Operating Procedure - 16

Decontamination of Submersible Pumps

Materials:

Field logbook

Personal protective equipment (PPE)

Polyethylene sheeting

Garbage bags

Nonphosphate detergent (e.g., Liquinox or Alconox)

Tap water

Deionized water

Two containers (e.g., garbage cans, buckets, plastic tubs)

Nylon brushes

Isopropanol

Spray bottles

Paper towels

Note: To limit the potential for cross-contamination between wells, wells should be pumped in the order of increasing constituent concentrations whenever possible. This SOP assumes that dedicated tubing is being used at each well. If dedicated tubing is not being used, the tubing should also be decontaminated using the following procedures.

Decontamination Procedure:

1. Use appropriate PPE as specified in the site-specific health and safety plan.
2. Prepare a decontamination area by spreading polyethylene sheeting on a firm, flat surface (if possible). Create a berm around the decontamination area to contain inadvertent spillage. A berm can be created by rolling under the edges of the polysheeting or by draping the plastic over a wooden frame, etc.
3. Place two clean containers (e.g., garbage cans, buckets, plastic tubs) on the polysheeting. Place tap water in one container with non-phosphate detergent. Place only tap water in the second container. The containers may also be lined with garbage bags.
4. If an oily film or residue is observed on the pump or leads when they are removed from the well, the pump should be sprayed with isopropanol to remove the oil and then wiped clean with paper towels before proceeding with Step 5 below. The oily rinsate should be contained in a separate container for proper disposal.
5. Place the pump and wire leads in the container of non-phosphate detergent and tap water and scrub the exterior of the pump with a brush. Circulate the soapy solution through the pump for at

least 5 minutes. Rinse the exterior of the pump and leads with additional tap water to remove excess soap (if necessary) before proceeding with Step 6.

6. Place the pump and leads in the container of tap water and run the pump for a least 5 minutes. Run water through the pump until all residual detergent has been removed. The soapy solution and rinse water should be changed when it becomes oily or too silty.
7. Remove the pump and leads from the rinse water. Spray off the pump thoroughly with deionized water and wipe it dry with clean paper towels. Wipe off the wire leads with a paper towel soaked with deionized water.
8. Wrap the pump and leads in plastic sheeting or a new plastic garbage bag to prevent possible contamination during transportation. Label the sheeting or bag with the date of decontamination for future reference.
9. Properly manage all PPE and decontamination rinsate in accordance with state and federal requirements (See SOP 26). The spent wash water and rinse water can potentially be placed in the facility's waste water treatment system. However, field personnel should obtain approval from facility personnel and from the local POTW.

Standard Operating Procedure - 17

Decontamination of Water Level Indicators

Materials:

Field logbook

Personal protective équipment (PPE)

Non-phosphate detergent (e.g., Liquinox or Alconox)

Deionized water

Isopropanol

Two buckets

Spray bottles

Paper towels

Note: To limit the potential for cross-contamination between wells, wells should be gauged in the order of increasing constituent concentrations whenever possible.

Decontamination Procedure:

1. Use appropriate PPE as specified in the site-specific health and safety plan.
2. If the groundwater is grossly contaminated (i.e., LNAPL or DNAPL is present), the tape should be pulled out of the well, NOT reeled up, and placed directly into a bucket of nonphosphate detergent and tap water. The tape and probe should be scrubbed with a brush to remove visible contamination. The tape and probe should then be rinsed in a second bucket of tap water before proceeding with Step 3. If persistent stains or oily films remain, apply isopropanol to a paper towel and wipe the tape and probe until clean.
3. Thoroughly wet a paper towel with deionized water from a spray bottle. Fold the paper towel over the tape and wipe it as the tape is reeled up.
4. The water level probe should also be sprayed with deionized water and wiped dry with a clean paper towel.
5. Place water level indicator in the clean carrying case or in a clean plastic bag to prevent contamination during transportation.
6. Properly manage all PPE, used paper towels, and decontamination rinsates in accordance with state and federal requirements (See SOP 26).

Standard Operating Procedure - 18

Decontamination of Interface Probe

Materials:

Field logbook

Personal protective equipment (PPE)

Nonphosphate detergent (e.g., Liquinox or Alconox)

Deionized water

Isopropanol

Two buckets

Spray bottles

Paper towels

Decontamination Procedure:

1. Use appropriate PPE as specified in the site-specific health and safety plan.
2. If the groundwater is grossly contaminated (i.e., LNAPL or DNAPL is present), the tape should be pulled out of the well, NOT reeled up, and placed directly into a bucket of nonphosphate detergent and tap water. The tape and probe should be scrubbed with a brush to remove visible contamination. The tape and probe should then be rinsed in a bucket of tap water before proceeding with Step 3. If persistent stains or oily films remain, apply isopropanol to a paper towel and wipe the tape and probe until clean.
3. Thoroughly wet a paper towel with deionized water from a spray bottle. Fold the paper towel over the tape and wipe it as the tape is reeled up.
4. The interface probe should be sprayed with deionized water and wiped dry with a clean paper towel.
5. Place the interface probe in the clean carrying case or in a clean plastic bag to prevent contamination during transportation.
6. Properly manage all PPE, used paper towels, and decontamination rinsates in accordance with state and federal requirements (See SOP 26).

Standard Operating Procedure - 19

Decontamination of Sampling Equipment

Materials:

Field logbook
Personal protective equipment (PPE)
Deionized water
10% nitric acid solution
Nylon brushes
Hand trowels, shovels, or spoons (stainless steel or Teflon)
Bowls (stainless steel or Teflon)
Hand Trowel (stainless steel or Teflon)
Containers (e.g., garbage cans, buckets, plastic tubs)
Nonphosphate detergent (e.g., Liquinox or Alconox)
Isopropanol
Aluminum foil
Polyethylene sheeting
Plastic garbage bags
Paper towels
Spray bottles
Duct tape

Note: All sampling equipment must be decontaminated before shipment to the office.

Decontamination Procedure:

1. Use appropriate PPE as specified in the site-specific health and safety plan.
2. Prepare a decontamination area by spreading polyethylene sheeting on a firm, flat surface (if possible). Create a berm around the decontamination area to contain inadvertent spillage. A berm can be created by rolling under the edges of the polysheeting or by draping the plastic over a wooden frame, etc.
3. Prepare a solution of nonphosphate detergent and tap water in a container.
4. Wipe sampling equipment with paper towels to remove residual soil or gross contamination. Heavy oils or grease may be removed with paper towels soaked with isopropanol.

5. Disassemble sampling equipment (e.g., split-spoon samplers and bailers). Wash equipment thoroughly in a nonphosphate detergent and hot tap water (if available) solution. Teflon bailers must be disassembled and the inside washed with a long-handled bottle brush or short-handled brush pulled through the bailer with rope.
6. Rinse the equipment with hot tap water (if available).
7. If the equipment will be used to collect samples for metals analysis, follow the tap water rinse with a 10% nitric acid solution rinse. Carbon steel equipment (e.g., bucket augers, split-spoons) should be rinsed with 1% nitric acid solution to reduce the potential for oxidizing the metal surfaces. Collect the nitric acid rinse in a separate bucket for proper disposal. Rinse the equipment with tap water.
8. Thoroughly rinse the equipment with deionized water.
9. Spray the equipment with isopropanol and allow to completely air dry. The solvent rinse must be collected in a separate bucket. Isopropanol is the recommended solvent for organic contaminants because it is readily available and is not a Department of Transportation hazardous material. However, other solvents (e.g., acetone, hexane, methanol) may be more effective in removing certain contaminants, such as oils or PCBs. Please note that many state programs and USEPA regions specify the solvents to be used for equipment decontamination.
10. Rinse the equipment with deionized water using at least five times the volume of solvent used in the previous step.
11. After the equipment has been allowed to completely air dry, each piece must be individually wrapped with aluminum foil (shiny side out), and then wrapped in plastic.

Note: Decontamination solvents may introduce contaminants to environmental samples. It is very important to ensure that the equipment has completely dried before use or storage.

12. After the final decontamination event on a project, label each piece of equipment with the date of decontamination, the initials of decontamination personnel, and the type of decontamination solutions used.
13. Note any discrepancies from standard decontamination procedures in the field logbook.
14. Field decontamination presents unique problems in disposal of decontamination solutions. The spent wash water and rinse water can potentially be placed in the facility's waste water treatment system. However, field personnel should obtain approval from facility personnel and from the local POTW. If no wastewater treatment system is present onsite, or if approval cannot be obtained from the facility and local POTW, the wash water should be containerized for offsite disposal in accordance with state and federal requirements. The volume of spent solvent generated during field decontamination should be minimal. Solvents should be collected in separate buckets and allowed to evaporate. See SOP 26 for information on managing investigation-derived wastes.
15. Paper towels soaked with solvent should be allowed to air dry and be disposed of with the general trash. Under no circumstances should any decontamination solution be disposed of on soil surfaces..

Standard Operating Procedure – 20

Sample Shipping Procedures

Materials:

Suitable shipping container (e.g., plastic cooler or lab supplied styrofoam cooler)

Chain-of-custody forms

Custody seals

WSP mailing labels

Strapping, clear packing, or duct tape

Ziploc® plastic bags

Knife or scissors

Permanent marker

Latex or nitrile gloves

Large plastic garbage bag

Wet ice

Bubble wrap or other packing material

Universal sorbent materials

Sample container custody seals (if required)

Federal Express form (with WSP account number)

Vermiculite (or commercially available cat litter)

Procedures:

1. For shipping purposes, samples are segregated into two classes; environmental samples and restricted articles (i.e., hazardous materials). Environmental samples can also be categorized based on expected or historical analyte levels (i.e., low or high). An environmental sample is one that is not defined as a hazardous material by the Department of Transportation (DOT, 49 CFR Part 171.8). The DOT defines a "hazardous material" as a substance which has been determined by the Secretary of Transportation to be capable of posing an unreasonable risk to health, safety, and property when transported in commerce, and which has been so designated. Any material of a suspected hazardous nature, previously characterized as hazardous, or known to be hazardous is considered a restricted article.
2. In general, the two major concerns in shipping samples are protecting the samples from incidental breakage during shipment and complying with applicable DOT and courier requirements for restricted article shipments.
3. Protecting the samples from incidental breakage can be achieved using "common sense." All samples should be packed in a manner that will not allow them to freely move about in the cooler or shipping container. Glass surfaces should not be allowed to contact each other. When

possible, repack the samples in the same materials that they were originally received in from the laboratory. Each container should be cushioned with plastic bubble wrap, styrofoam, or other nonreactive cushioning material. Shipping hazardous materials should conform to the packaging, marking, labeling, and shipping instructions identified in 49 CFR Parts 172 & 173.

4. Environmental samples shall be packed for shipment using the following procedures:
5. Line the shipping container with a large, heavy-duty plastic garbage bag. Place universal sorbent materials (e.g., sorbent pads) between the cooler and the heavy-duty plastic bag. The amount of sorbent material should be sufficient to absorb the volume of wet ice and aqueous samples. If using a plastic cooler, securely tape the drain plug closed on the outside of the cooler.
6. Place 2-4 inches of bubble wrap or other packing material inside the heavy-duty plastic bag in the bottom of the cooler.
7. The sample packer should wear latex or nitrile gloves when handling the samples during the packing process.
8. Place the bottles in the cooler with sufficient space to allow for the addition of more bubble wrap or other packing material between the bottles. Large or heavy sample containers should be placed on the bottom of the cooler with lighter samples (i.e., VOAs) placed on top to eliminate breakage.
9. Place the "wet ice" inside two sealed heavy-duty zipper-style plastic bags and package the bags of ice on top of or between the samples. Pack enough ice in the cooler to chill the samples during transit. If the cooler is shipped on a Friday or Saturday for Monday delivery, double the amount of ice placed in the cooler (Monday delivery should be used only as a last resort). Fill all remaining space with bubble wrap or other packing material. Securely close and seal with tape the top of the heavy-duty plastic bag.
10. Place chain-of-custody form (and, if applicable, CLP traffic reports) into a Ziploc® plastic bag and affix to the cooler's inside lid, then close the cooler. Securely fasten the top of the cooler shut with tape. Place two signed and dated chain-of-custody seals on the top and sides of the cooler so that the cooler cannot be opened without breaking the seals.
11. Once cooler is sealed, shake test the cooler to make sure that there are no loose sample containers in the cooler. If loose samples are detected, open the cooler and repack the samples.
12. Using clear tape, affix a mailing label with WSP's return address to the top of the cooler.
13. Ship samples via priority overnight express to the contracted analytical laboratory for next morning delivery. If applicable, check the appropriate box on the airbill for Saturday delivery.
14. Declare value of samples on the shipping form for insurance purposes. The declared value should reflect the cost to recollect the samples.
15. Record the tracking numbers from the Federal Express forms in the field notebook and on the chain of custody form. Also, retain the customer's copy of the Federal Express airbill.

Hazardous materials should be packed according to the above procedures with the following additions:

1. Place samples in individual Ziploc® plastic bags and secure with a plastic tie or tape.

2. Place samples in paint cans in a manner which would prevent bottle breakage (i.e., do not place glass against glass).
3. Place vermiculite or other absorbent packing material in the paint can around the samples. The amount of packing material used should be sufficient to absorb the entire contents of the sample if the container is broken during shipment.
4. Secure a lid to the paint can with can clips and label the outside of the can with sample numbers and quantity. Mark the paint can with "This End Up" and arrow labels that indicate the proper upward position of the paint can.
5. Package the paint cans in DOT-authorized boxes or coolers, with appropriate DOT shipping labels and markings on two adjacent sides of the box or cooler.
6. Ship the restricted articles via overnight courier following the courier's documentation requirements. A special airbill must be completed for each shipment. Retain a copy of the airbill for WSP records and tracking purposes, if necessary.

Standard Operating Procedure – 21

Field Quality Assurance/Quality Control Samples

Materials:

Field logbook

Personal protective equipment (PPE)

Sample containers

Sample labels

Clear tape

Laboratory analyte free water

Clean or dedicated sampling equipment

Procedure:

1. Use appropriate PPE as specified in the site-specific health and safety plan.
2. Select the appropriate glassware for the field Quality Assurance/Quality Control (QA/QC) samples. Refer to the WSP Standard Operating Procedure for Sample Container, Preservatives, and Holding Times to determine the appropriate bottles to use.
3. Field QA/QC samples include the following:
    a. trip blanks
    b. duplicate samples
    c. equipment blanks
4. Trip blanks should be provided by the analytical laboratory for all projects where samples are being collected for analysis of volatile organic compounds (VOCs). Trip blanks should accompany the sample bottles from the analytical laboratory to the site, accompany the sample containers at all times during the sampling event, and return to the laboratory with the sample containers. One trip blank should be submitted to the analytical laboratory with each shipment containing samples for VOC analysis. The trip blank should be analyzed only for VOCs.
5. One duplicate sample should be collected for every 20 samples of each matrix (e.g., soil and groundwater) collected during each sampling event. Duplicate samples of soil and other solid matrices (sediments) should be collected by dividing the sample material in half and alternately filling the two sample bottle sets. Duplicate samples of groundwater and other aqueous matrices should be collected by alternately filling the two sample bottle sets from the same sampling vessel (e.g., bailer). The appropriate SOP should be followed for the collection of each sample type (soil, groundwater, sediment, sludge). Duplicate samples should be analyzed for all the analytes that are being analyzed for during the sampling event.
6. One equipment blank should be collected in the field at a rate of one per type of equipment per decontamination event not to exceed one per day. If dedicated sampling equipment is used, the equipment blanks should be prepared in the field before sampling begins. If field decontamination

of sampling equipment is required, the equipment blanks should be prepared after the equipment has been used and field-decontaminated at least once. Equipment blanks should be prepared by filling or rinsing the precleaned equipment with analyte-free water and collecting the rinsate in the appropriate sample containers. The samples should be labeled, preserved, and filtered (if required) in the same manner as the environmental samples. Equipment blanks should be analyzed for all the analytes for which the environmental samples are being analyzed. Decontamination of the equipment following equipment blank procurement is not required.

7. All QA/QC samples should be submitted to the analytical laboratory with unique sample numbers. Therefore, the QA/QC samples should be labeled as separate environmental samples following the same numbering scheme used during that particular sampling event. However, the QA/QC samples should be clearly identified on WSP's copy of the chain-of-custody form and in the field logbook.

Standard Operating Procedure - 22

Soil Head Space Screening (Field Technique)

Materials:

PPE

Field logbook

Photoionization detector (PID) and/or Flame Ionization Detector (FID)

Aluminum foil

Clear 8-oz to 16-oz glass large-mouth containers with lids

Stainless steel spoon

Zipper-style plastic bags

Procedure:

1. Use appropriate PPE as specified in the site-specific health and safety plan.
2. Check PID to ensure that it is working properly.
3. Using WSP's standard operating procedure (SOP) for collecting soil, sludge, or sediment, half-fill a clean glass jar with sample. Place a piece of aluminum foil over the top of the jar and tightly seal the jar. Label the jar indicating the sampling location, depth, and date. Store the jar upside down until the sample is analyzed.
4. If jars are not available, collect the sample using a zipper-style plastic bag (e.g., Ziploc®). Seal and label the bag as specified in item 3.
5. Shake the sample vigorously for approximately 15 seconds.
6. If necessary, warm the sample to room temperature (70*F) by placing the jar in a heated room or vehicle. This step is very important when the ambient temperature is below 32*F.
7. After waiting approximately 15 minutes, carefully unscrew the lid of the jar without disturbing the aluminum foil. Pierce a hole through the aluminum foil using the tip of the PID. If using zipper-style bags, open the bag slightly and place the tip of the probe into the opening. Do not insert the probe into the soil and avoid the uptake of water droplets.
8. Following probe insertion, record the highest meter response. Using the foil seal/probe insertion method, maximum response should occur between 2 seconds and 5 seconds. Erratic PID response may result from high organic vapor concentrations or elevated headspace moisture. If these conditions exist, the headspace data should be qualified or discounted.
9. Record the sample location, depth, soil texture (i.e., clay or sand), and PID reading in the field notebook. Also record the ambient temperature, humidity, and whether moisture was present in the jar or plastic bag. These points are important because on very cold days volatilization of organic compounds is reduced and water vapor present in the jar may cause the PID to give a false reading. Be consistent in your procedure and in your recording of the data.

10. Duplicate 10 % of the headspace samples by collecting two samples from the same location and following items 2 through 9 above. The headspace screening data from both jars should be recorded and compared. Generally, replicate values should be consistent to plus or minus 20%.
11. Samples collected for headspace screening should not be retained for laboratory analysis. Dispose of the soil and jar appropriately

Standard Operating Procedure – 23

Underground Utility Locating

Application:

The purpose of this procedure is to ensure that all required and appropriate procedures are followed to locate and mark subsurface utilities (e.g., electrical lines, natural gas lines, communication lines) before initiating any intrusive field activities (e.g., drilling, test pits, trenching). Compliance with this procedure is mandatory before intrusive work can be conducted on a WSP project. This procedure is intended to allow the work to proceed safely and will minimize the potential for damaging underground utilities. Intrusive work includes all activities that require WSP's employees or their subcontractors to penetrate the ground surface. Examples of intrusive work include, but are not limited to probing, drilling, injection, test pit excavations, trenching, and remedial excavations.

Materials:

Record of the communication utility locating form (Attachment 1)

Field logbook

Wooden stakes

Spray paint

Flagging tape

As-built drawings for sub grade utilities (if available)

Hand auger or post-hole digger

Hand-held magnetometer or cable locator (optional, if and only if private utility locator has cleared the area and personnel have been properly trained in the use of the equipment)

Procedure:

Pre-site Mobilization

1. Gather the necessary information to complete the record of communication utility locating form (Attachment 1).
2. Contact the state utility locating service (e.g., One-Call, Miss-Dig). It is imperative to contact the locating service with sufficient lead-time to allow all utility providers to visit the site location. In each case, the state utility locating service will provide the caller with a legal dig date. Under no circumstances will intrusive work begin before the legal dig date provided by the call center. The telephone numbers for the locating service in selected states are listed in Table 1. However, the telephone number is typically listed in the area Yellow Pages. Provide the utility locating service with any information they request concerning the site and work activity in order to locate utilities at the site. Several states, including California, require that the proposed drilling locations be marked with white spray paint before contacting the locating services. The following information provided by the locating service should be documented in a record of communication utility locating form (Attachment 1): utility providers that will be contacted, and a utility clearance ticket

number. The ticket number will be used by the various utility companies to reference the clearance request and to contact the caller with clearance verifications (see note below).

Note 1: Generally, the public utility companies will mark underground lines up to the private property boundary. However, you should request that the utility companies mark their utilities in the work areas on the site. If the utility companies will not provide that service, a private utility locating service MUST be contracted.

Note 2:Some utilities (e.g., sewer, water, cable TV) may not be included by the State locating service. The State locating service will provide you with a list of utilities that will be notified based on the information provided regarding the sites location. Compare this list with utilities generally expected at all sites (e.g., sewer, water, gas, communication, electric). If any expected utilities are absent from the contact list, you MUST contact the utilities directly for clearance before the start of intrusive activities. Record all contacts on the utility locating record of communication form.

3. Identify a site contact familiar with the utilities on the property (e.g., plant manager, facility engineer, maintenance supervisor), and provide this individual with a site plan showing the proposed locations of all soil borings, monitoring wells, test pits, and other areas where intrusive activities will be conducted. Ask the site contact for all drawings concerning underground utilities in the proposed work areas.
4. No intrusive work should be done before the legal dig date provided by the State utility locating service. No intrusive activities should be conducted along or near public right-of-ways until all utilities have been marked and visually verified in the area of investigation. In addition, NO field activities shall be conducted on private property unless the State locating service or a private utility locating service has confirmed the presence or clearance of onsite utilities.

Site Mobilization

1. Locate all proposed drilling and trenching locations, both onsite and offsite, with spray paint, stakes, or other appropriate markers.
2. Verify that ALL utility companies listed by the municipal locating service, and any contacted directly by WSP, have either marked the underground lines in the specified work areas or have responded with "no conflict." Document on the utility record of communication form as each utility mark is visually confirmed.

   Note: When receiving verbal clearances by telephone from utility companies, or their subcontractors, it is imperative that you verify which utilities are being cleared, particularly when dealing with subcontractors that may be marking more than one utility.
3. Review all available as-built utility diagrams and plans with the site contact to identify potential areas where underground lines may be present. The review should confirm the locations marked by the locating services and identify utilities that may have been omitted by the locating services. If the as-built drawings do not confirm utilities marked by the locating services, follow instructions in Section 6. If possible, obtain a copy(s) of the utility plans for future reference in the field.

   Conduct a site walk with the site contact. During the site walk, attempt to obtain a general knowledge of the types of utilities present in the work areas. Furthermore, survey your surroundings to identify features that require electricity (e.g., parking lot lights, pad-mounted transformers) or suggest the presence of underground utilities, such as linear depressions in the ground. Check these items against the utility locating record of communication form checklist. For

example, check to see whether major electrical lines are aboveground, or locate underground sewer lines by using the locations of manholes and storm water grates. Keep in mind that many sewer lines can be offset from catch basins.

4. A minimum of 4 feet clearance should exist between utilities and proposed drilling locations, and a minimum of 6 feet between utilities and proposed trenching locations. A minimum distance of 15 feet should be maintained by heavy equipment (e.g., excavator buckets, drill rig towers and rods) from overhead power lines. A safe distance of 25 feet should be maintained from high tension overhead power lines. In the event that work must be conducted within 25 feet of high tension wires, the lines should be wrapped and insulated by the local utilities. If a utility conflict is identified, adjust the proposed location(s) using the criteria given above. These minimum distances should be increased whenever possible to offer additional assurance that utilities will not be encountered.

5. A private utility locating service MUST be used for work on private property in cases where the public utility locating service does not mark utilities on the subject property. It is NOT ACCEPTABLE to rely on as-built drawings or verbal utility clearances. A private locator may not be necessary in rare instances; however, these cases must be discussed with the project manager AND a partner or executive partner of WSP before work may proceed.

   A listing of several private subsurface utility locating firms is provided in Table 2. In addition, a hand-held magnetometer or magnetic-cable locating device can be used to augment, but not replace, clearance for each work area. Use of this equipment is restricted to employees with proper training on the use of hand-held utility locating equipment. Proper training is defined as having working knowledge of the manufacturer's operating procedures, and the completion of at least one successful location under the supervision of a qualified person.

6. In some cases, state and private locating services may not be able to identify all utilities. In areas where uncertainty still exists concerning the presence of underground utilities after clearance by state and private locating services, a hand auger or post-hole digger can be used to probe the shallow subsurface before using any heavy equipment (drill rig, backhoe). The probe hole should be advanced a minimum of 4 feet below ground surface at each proposed drilling or excavation location. A sufficient number of probe holes should be completed so that the area is cleared for the proposed intrusive activity. For drilling, a minimum of three holes installed in a triangular pattern should be advanced at each location. The use of hand digging methods in NO WAY replaces the need State and private utility locating services. Hand digging techniques should only be employed if uncertainty regarding the location of underground lines still exists after clearances by the State locating service and a private locating service.

7. Discuss the site conditions with the subcontractor and recommend that care be used at the start of the intrusive activities. Field personnel should always consider the presence of unidentified utilities at each work area. In addition, field personnel have the authority and responsibility to postpone intrusive activities if insufficient information, as stipulated in this SOP, is available, or if onsite reconnaissance identifies inconsistencies in the findings of utility locators. In these instances, field personnel should contact the project manager or a member of the health and safety committee, and an executive partner or partner of WSP before proceeding with the proposed work. The first priority on every project is to ensure that the work is conducted safely.

   Again, it is the requirement of this SOP to obtain site utility clearances from the State utility locating service. If the State locating service does not provide onsite (i.e., work area) utility clearance, a private locating service must be contracted to clear the work areas before digging,

drilling, or probing begins. Although certain instances and site conditions may appear to allow intrusive work without prior clearance, ALL deviations from this SOP MUST be approved by the project manager and a partner or executive partner BEFORE beginning intrusive work.

8. If the scope of the intrusive activity locations changes, the scope of intrusion expands or includes a new onsite or offsite area(s), review the existing information to determine whether the area(s) can be safely cleared of all potential underground utilities. If necessary, contact the state locating service and request another clearance for the new area(s) of investigation and retain a private locator in accordance with Item 5 above. Remember, the new request will provide a new legal dig date before which NO INTRUSVE WORK CAN BEGIN. Additionally, if a clearance ticket will expire while the work is ongoing (typically after 14 days), a new clearance must be requested at before the first ticket expires so that work can continue uninterrupted. Refer to the communication utility locating form for the legal dig date time frame required by the State locating service.

PUBLIC and PRIVATE UTILITY LOCATING FORM

Project:_____ Project Manager:_____

Project Number:_____

Project Start Date:_____ Duration of Project:_____

Project Location (Site Address):_____

Project Site Description (complete the following with all information available *before* calling):

Work Being Done For: (*Company or Individual Name*): _____

State: _____County:_____ City/Place: _____

Street: _____(*Only one street per ticket*) Zip Code:_____

Nearest Intersecting Street: _____

Lat/Long:_____Parcel/Tax map ID:_____

Description of the area to be marked *(Provide the following: Street working on, which side of street, how far in which direction from nearest intersecting street; etc.)*:

_____

_____

Locations for proposed borings or digging identified with paint and/or stakes? (circle one): YES NO N/A

NOW, MAKE THE CALL:

Call Placed to Phone No.: _____

Date of Call:_____Time of Call:_____a.m. / p.m.

Ticket No. Assigned to Location Request:_____

Assigned Legal Dig Date: ___ / ___ / ___ Assigned Legal Dig Time: ____ AM / PM

The Following Table Must Be Completed Before Work Can Begin:

CALL BACK/FAX BACK INFORMATION RECORD						
	Gas	Communication	Electric	Water	Sewer	Other
Responsible Company (provided by One-Call operator)						
Date Notified						
Time Notified						
Notified By						
Phone Number						
Marks Complete						
No Conflict						
No Facilities						

Calls Made By:_____ Form Completed By:_____

Project Manager Notified of Results (initial if completed):_____

Onsite Underground and Overhead Utility Clearance Checklist

Visual Confirmation of Marked Public Utilities:

Utility Type	Visual Cues	Marks Confirmed (initial)	No Markings Seen (initial)
Water	Blue Markings, fire hydrant, manholes; water meter, sprinkler heads, AST, hose bib		
Gas	Yellow Markings, gas meter, manholes; yellow bollards		
Electric	Red Markings, parking lot lights, overhead lines (telephone poles), underground vaults, manholes; conduit on buildings		
Sewer (sanitary/storm)	White or Blue Markings, underground vaults, manholes, drain grates		

Communication	Red or White Markings, red bollards, telephone poles; manholes; conduit on buildings		

Visual Confirmation of Marked Private Utilities (at onsite drilling/digging locations):

Utility Type	Visual Cues	Marks Confirmed	No Markings Seen (initial)	Not Applicable (initial)
Water	fire hydrant, manholes; water meter, ASTs, interior connections, hose bib, valve box			
Irrigation	sprinkler heads, hose bibs			
Gas	gas meter, manholes; yellow bollards, interior connections, valve box			
Electric	parking lot lights, interior connections, overhead lines, underground vaults, manholes, transformers/switchgear; conduit on buildings			
Sanitary/Storm/ Septic System	underground vaults, manholes, drain grates, leach field, sand mound, no evidence of sanitary sewer (for septic system			
Production Equipment	USTs (fill pipes and vent pipes), ASTs (overhead and underground pipelines), manholes/valve pits; pump islands			
Communication	Red/orange bollards, telephone poles, interior connections; manholes; conduit on buildings			

If any Utilities have "No Marking Seen" checked, private locating must be conducted to clear each drilling/digging area.

Site Visit Made By:_____

Form Completed By:_____

Standard Operating Procedure – 24

Soil Sampling Using GeoProbe® System or Equivalent

Application:

To perform depth-discrete soil sampling with 2-foot or 4-foot long samplers using hydraulically-driven soil sampling equipment (GeoProbe® System or Equivalent).

Materials:

Stainless steel soil sampler (2-foot or 4-foot long)

Clear acetate liners

Tape measure or expandable ruler

Utility knife

Photoionization detector (PID)

Stainless steel spoons

Aluminum tray or stainless steel mixing bowla

Nitrile or latex gloves

Field notebook

Procedure:

1. Calibrate the PID in accordance to the manufacturer's instructions. Decontaminate all down-hole sampling equipment and the utility knife, spoons, and mixing bowl per SOP 19 before initiating any boring activities. Ensure that the location is clear of all underground utilities and pipelines.
2. Attach a decontaminated 2-foot or 4-foot long stainless steel sampler fitted with a new, clear acetate liner and a decontaminated removable cutting shoe to small-diameter rods. Lower the stainless steel sampler to the top of the desired sampling depth.
3. Advance the stainless steel sampler through the desired sample interval. Record in the dedicated field notebook the interval through which the sampler was pushed.
4. After the sampler has reached the desired depth, retrieve the sampler by first removing the rods and then disconnecting the sampler. Remove the cutting shoe and acetate liner containing the soil column from the sampler. Measure the length of the material recovered relative to the interval the sampler was advanced, and record this information in the field notebook.
5. Cut the acetate liner using a utility knife to expose the recovered soil. Quickly scan the recovered soil with the PID and if necessary, immediately collect samples for VOC analysis. If the plan indicates the collection of samples for headspace analysis, collect this sample after obtaining the sample for VOC analysis per SOP 22. Record the PID readings in the field notebook.

6. For VOC samples, transfer soil directly from the acetate liner into the sample containers with a clean, stainless steel spoon. Fill the VOC sample container with a representative sample from the entire length of the recovered sample core, or other designated sample intervala. Fill the VOC container completely, leaving no headspace.
7. Describe the recovered soil using the Unified Soil Classification System or standard geological descriptions. Record the sample description in the field notebook.
8. If it is necessary to mix the sample, transfer the soil from the acetate liner to a clean aluminum tray or decontaminated stainless steel mixing bowl with a decontaminated stainless steel spoonb.
9. Examine contents of the tray/bowl and remove rock fragments and organic debris, such as roots, grass, and woody material, with the stainless steel spoon. Use the same spoon to chop apart clumps of dirt and mix the contents of the tray to a homogeneous particle size and soil texture. Transfer the tray/bowl contents to the appropriate sample containers using the stainless steel spoon.
10. The sample container(s) should be sealed, labeled, and placed in a cooler with ice or freezer packs to maintain 4° Celsius for shipment to the analytical laboratory.
11. Complete the chain-of-custody form with the appropriate sampling information.
    a. NJDEP's Field Sampling Procedures Manual requires the collection of soil samples for VOC analysis from the 0.5-foot interval that exhibits the highest reading during the field (PID) screening.
    b. U.S. Environmental Protection Agency (EPA) Region 4 requires a glass bowl for homogenizing soil for sample collection.

Standard Operating Procedure – 25

Groundwater Sampling Using Geoprobe® System or Equivalent

Application:

To perform groundwater sampling using hydraulically-driven screen point sampling equipment (GeoProbe® System or Equivalent).

Materials:

Stainless steel probe rods with treads sealed with Teflon® tape or O-rings

Stainless steel screen point sampler

Stainless steel mini-bailer

Teflon®-coated stainless steel wire or thin nylon line

Polyethylene tubing (3/8-inch) fitted with a stainless steel check valve

Silicone tubing

0.45-micron filter

Peristaltic pump

Sample bottles, labels, indelible markers, and clear tape

Nitrile or latex gloves

Procedure:

1. Decontaminate all down-hole equipment before conducting sampling activities at each location. Ensure that the sampling location has been cleared of all underground utilities.
2. Drive the stainless steel point sampler into the subsurface material. The design of the sampler should allow the screen to remain retracted within the probe rods until it is driven to the appropriate sampling depth.
3. After reaching the desired depth, pull back on the stainless steel sheath to expose the screen. The point on the probe rods will be displaced and is not recoverable.
4. Purging is not required for probes that are sealed and opened at the target depth for sample collection. Exposed probes that are driven through the soil to the desired water sample depth must be purged of a minimum of three probe-rod volumes of water before sampling is conducted.
5. Groundwater samples can be collected using a mini-bailer lowered on Teflon®-coated stainless steel wire or nylon line inside the probe rods. Another method of sample collection involves the use of a clean section of 3/8-inch polyethylene tubing fitted with a stainless steel bottom check valve. The polyethylene tubing is inserted down the probe rods to the desired sampling depth. Oscillate the polyethylene tubing up and down to drive a column of water to the surface. A peristaltic pump may be attached to the sample tubing and used to pump water to the surface. The peristaltic pump should not be used to collect samples for VOC analysis.

6. Immediately collect samples for VOC analysis, if required. Transfer the groundwater directly from the sampling equipment (mini-bailer, polyethylene tubing) to the sample containers. If analyzing for dissolved metals, the sample must be filtered in the field. See note below with regards to field filtering of metal samples.
7. Seal and label each sample container and place in a cooler with ice or freezer packs to maintain 4° Celsius for shipment to the analytical laboratory.
8. Complete the chain-of-custody form with appropriate sampling information.
    a. Where samples are collected from depths greater than 15-20 feet below the water table, a water level indicator may be inserted into the rods before exposing the screen to determine whether water is entering through the rod joints or disposable probe point.

Field Filtering of Metal Samples:

1. Assemble peristaltic pump per operating manual instructions that accompany the pump. Silicone tubing is generally used though the head of the pump.
2. Attach polyethylene tubing to the inflow end of the silicone tubing. The polyethylene tubing should be long enough to extend to the bottom of the screen point. Attach a clean filter to the outflow end of the silicone tubing.
3. Turn on the pump and slowly draw the water from the sampling equipment, through the pump and filter, and into the sample container. If sediment is visible in the sample container, filter break-through has occurred and the sampling and filtering process will need to be repeated.
4. Disassemble the pump head and discard the tubing and filter.

Standard Operating Procedure – 26

Managing Investigation Derived Waste

Application:

The purpose of this SOP is to provide instructions for handling, storing, and sampling Investigation Derived Waste (IDW) pending disposal. All IDW should be handled as hazardous waste unless information exists which would allow it to be classified as non-hazardous waste. IDW generated during a Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) response action must be managed in compliance with applicable or relevant and appropriate requirements (ARARs) to the extent practicable and with applicable requirements of the CERCLA offsite policy. (EPA Guidance Document OERR Directive 9345.3-02)

IDW includes soil cuttings, development water, purge water, drilling fluids, decontamination fluids, personal protective equipment, and sampling equipment.

Materials:

Non-Hazardous and Hazardous Waste Labels

Investigation Derived Waste Log (Figure 1)

Permanent Ink Marking Pen Paint Stick/Pen

Sampling Equipment (Refer to Sampling SOPs)

Sample Jars

Chain of Custody Forms

Cooler

Procedure:

1.1.12 Hazardous IDW

1. All IDW should be handled as hazardous waste unless information exists which would allow it to classified as non-hazardous waste. New or existing site data (i.e., soil and groundwater results) and generator knowledge can be used to classify the IDW.

- If site data or generator knowledge indicates that the IDW is determined to be hazardous the following procedures will apply:
- The IDW must be placed in DOT approved containers (55-gallon drum, roll-off container, or temporary storage tank).
- The containers must remain closed except when adding, sampling, or inspecting the material.
- All containers must be labeled with the words "Hazardous Waste".
- An accumulation start date and the contents of the container must be included on the label.

- Investigation Derived Waste Logs (Figure 1) must be completed before leaving the site. One copy of the log should be presented to the site contact and the original provided to the project manager. Once the material has been removed from the site, the IDW log should be stamped "Removed" and placed in the project file.
- The IDW containers must be stored in a secure onsite location (facility hazardous waste storage area if one exists).
- Disposal of the IDW must be completed within 90 days of the date the waste was generated. If the facility is a small quantity generator, 180 days is allowed for shipment of the waste offsite.
- Onsite disposal may be allowed or appropriate under certain conditions. Refer to OERR Directive 9345.3-02 for guidance, especially for CERCLA sites.
- WSP personnel should notify the site contact that weekly inspections of the IDW must be conducted and documented.
- WSP personnel should also instruct the site contact that this waste must be included in the facilities annual or biannual reports.

2. If the IDW is presumed to be hazardous and sampling is required to confirm its classification, it should be labeled Hazardous Waste-Pending Analysis. The waste should be sampled before leaving the site (See sampling SOPs). It should be noted that EPA methods 8260 and 8270 may be more cost effective than running the full Toxicity Characteristic Leaching Procedure (TCLP) scan. TSD Facilities will usually specify the required analysis for their waste profiles.

1.1.13 Non-Hazardous IDW

1. If information exists to classify the IDW as non-hazardous waste, the following procedures can be implemented:

1.1.14 Soil Cuttings

- Spread around the borehole or other onsite location with the approval of facility personnel
- Place back in the boring
- Containerize and dispose offsite

1.1.15 Groundwater

- Pour onto ground next to well to allow infiltration
- Containerize and dispose offsite
- Discharge to POTW with approval of facility personnel
- Discharge to onsite wastewater treatment plant with approval of facility personnel

1.1.16 Purge water from onsite well development

- Collect purge water from well development
- Containerize water in steel drums
- Profile waste water through laboratory chemical analysis
- Discharge to onsite wastewater treatment plant with approval of facility personnel

1.1.17 Decontamination Fluids

- Pour onto ground (from containers) to allow infiltration
- Containerize and dispose offsite
- Discharge to POTW with approval of facility personnel
- Discharge to onsite wastewater treatment plant with approval of facility personnel

1.1.18 PPE

- Double bag and deposit in site dumpster
- Containerize and dispose offsite

If the IDW is containerized and is non-hazardous, the following procedures will apply:

- The non-hazardous IDW must be placed in DOT approved containers (55-gallon drum, roll-off container, or temporary storage tank).
- The containers should remain closed except when adding, sampling, or inspecting the material.
- All containers must be labeled with the words "Non-Hazardous Waste".
- An accumulation date and the contents of the container should be included on the label.
- Complete the IDW log (Figure 1). One copy of the log should be presented to your site contact and the original should be given to the project manager.
- The IDW containers must be stored in a secure onsite location.
- Arrangements for disposal should be completed within 90 days of the accumulation start date.

Investigation Derived Waste Log

Date: _____

Site Information

Site Name: _____ Site EPA ID #: _____

Site Contact: _____ Site Address: _____

Contact Telephone No: _____

Waste Identification

Type of Waste Generated (check one of the following):

☐ Soil Cuttings ☐ PPE ☐ Decontamination Water

☐ Groundwater ☐ Storm Water ☐ Drilling Fluids

☐ Other (Describe): _____

Field Activities that generated the Waste:

☐ Soil Borings ☐ Well Sampling ☐ Well Installation

☐ Decon ☐ Excavation ☐ Pumping Tests

☐ Other (Describe): _____

Generation Date: _____ ***90-Day Deadline:*** _____

Quantity of Waste Generated and Container Type: _____

_____

_____

Storage Location: _____

Waste Identification (Check One of the Following);

☐ Non Hazardous Waste (pending analysis)
☐ Non Hazardous Waste (based on site information or generator knowledge)
☐ Hazardous Waste (pending analysis)
☐ Hazardous Waste (based on site information or generator knowledge)

If generator knowledge or site information was used for identification, explain: _____

_____

_____

Type of Label Applied to Container: ☐ Non Haz ☐ Hazardous ☐ PCB ☐ Used Oil

WSP Information (Note: One copy to site contact - the original in project file)

Personnel/Contact: _____ Project No.: _____
Telephone _____

Standard Operating Procedure – 27

Soil Sampling Using a Rotosonic Sampler

Application:
To perform soil sampling with a rotosonic drill rig sampler advanced ahead of the temporary casing.

Materials:

Rotosonic samplers
Rotosonic sample bags (plastic)
Knife
Photoionization detector (PID)
Ziplock plastic bags (quart size)
Nitrile or latex gloves
Sample containers
Field Notebook
Marker

Procedure:

1. Decontaminate all downhole drilling and sampling equipment in accordance with the appropriate procedures before initiating any intrusive work.
2. Ensure that the temporary rotosonic casing has been advanced to immediately above the desired sample depth and loose soil in the bottom of the borehole has been removed.
3. The drillers will attach a decontaminated rotosonic sampler to the drilling rods, lower it to the bottom of the borehole, and advance the sampler through the soil using the rotosonic rig. (Note: the standard rotosonic sampler is capable of collecting a 10-foot long continuous core although 5-foot samplers may also be available. It may be desirable to advance the sampler less than 10 feet to avoid penetrating relatively thin confining layers.)
4. After the sampler has penetrated the desired sample interval, the drillers will retrieve the sampler and disconnect it from the drilling rods.
5. The drillers will place a 4-inch diameter plastic bag over the bottom end of the sampler and vibrate the rotosonic drill head until the sample is extruded from the core barrel and fills the bag. After filling, remove the bag and place it on plastic sheeting for examination and screening. Note the orientation of the sample (i.e., which end is "up") and the depth interval. Be sure the examination/screening area is far enough away from the drilling rig exhaust pipe or other potential sources of contaminants that could affect the screening results.
6. Wearing new gloves, cut the sample bag lengthwise using a decontaminated knife. Immediately scan the recovered soil material with a PID at approximately 2-foot intervals and record the readings in the field notebook.
7. Collect samples for soil headspace analysis using the procedure described in WSP's SOP 22 or other approved method/guidance. Record all headspace readings in the field notebook.a
8. If soil samples are being collected for VOC analysis, immediately transfer soil directly from the recovered core into the appropriate sample container(s). Collect the sample from the desired depth based on the sampling plan or field screening results. Label the container(s) with the

sample name, depth, date and time of collection, sampler name, and analysis requested. Immediately place the containers in a cooler with ice to maintain a temperature at 4oC.

9. Collect samples for other parameters, as needed, in accordance with the sampling plan.
10. Describe the recovered soil using the Unified Soil Classification System or standard geological descriptions, and include PID measurements, sample number(s) and depth(s), and observations such as staining or odor. Write all descriptive information in the field notebook.
11. Complete the chain-of-custody form with the appropriate sampling information.

Standard Operating Procedure – 28

Sampling of Private Water Supply Wells

Scope:
This procedure describes the method for collecting representative groundwater samples from a domestic or other private water supply well.

Materials:

Business cards
Field notebook
Temperature, pH, and conductivity meter(s)
Sample containers
Cooler
Labels
Indelible markers
Nitrile or latex gloves
Chain of custody forms
Bucket or pail (1 or 2-gallon)
Paper towels or Kimwipes

Pre-Sampling Activities:

1. Verify the location of the well to be sampled and analytical parameters as specified in the sampling plan.
2. Contact the well owner and explain the scope of work. If no information has been obtained about the well, try to obtain information from the owner regarding the exact well location, well construction details, static water depth, well depth, casing diameter, well yield, date drilled and drilling company, previous testing, presence of water treatment components, resource aquifer, and well use. Locate the well on a site or USGS topographic map for future reference.
3. If insufficient well information is obtained from the owner, consider performing a water well survey to obtain the information.

Sampling Procedures:

1. Prepare the field notebook with description of site, well location and owner, weather conditions, sampling personnel, and other relevant observations.
2. Inspect the well to verify construction and other information concerning the well.
3. Identify a cold water tap or spigot for sample collection. The sampling point should be as close to the well as possible, and before the water passes through any treatment systems, heating unit, or storage system. If a suitable sampling point is not available, try to find another well at the site or take steps to minimize the impact of any potential interferences to the sample. Document in the field notebook whether you can or cannot collect a water sample before any treatment or storage components.
4. If sampling from a tap or faucet, try to remove any aerators, filters, or other devices from the tap before purging. If the sampling point is outside, consider removing any hoses connected to the spigot. (However, hoses may be allowed to remain attached during purging to divert large volumes of water from buildings, septic systems, etc).

5. Start to purge the system by running water from a tap or outside spigot. Use a small bucket or pail and watch to determine the purge rate for the system. If the water sample is to be collected from a tap located after a pressurization or holding tank, a sufficient volume should be pumped from the well to allow for the complete exchange of water into the holding tank and the point where the sample is to be collected.
6. Geochemical measurements (pH, conductivity, and temperature) of the water should be collected at regular intervals (every 1-2 minutes) after the start of purging. Record all measurements in the field notebook.
7. Review the geochemical measurements to ensure that readings have stabilized (within 10% of the previous reading). If the geochemical measurements have not stabilized, continue to purge the well until the measurements do not vary more than 10% between two successive measurements (if possible).
8. Collect the groundwater samples after purging is complete. While collecting samples, reduce the water flow to a thin stream. The flow rate should be high enough to deliver a smooth stream of water without splashing but low enough to permit filling of the sample bottles without turning the valve down during sampling. Collect the samples in accordance with the procedures described in WSP's SOP 2 – Sample Containers Preservatives and Holding Times. Label each container with the site name, sample name, date and time of collection, samplers, preservatives (if added), and analyses. Place the sample containers in a rigid cooler with ice maintained at a minimum of 40C.
9. Decontaminate all field meters and other non-disposable equipment, and properly dispose of any used paper towels, gloves, etc.
10. Complete chain-of-custody forms with appropriate sampling information.

RECORD OF COMMUNICATION
UTILITY LOCATING FORM

Date of Call: _____/ _____/ _____ Time of Call: _____a.m. / p.m.

Name of Utility Service Protection Center Called: _____

Call Placed to Phone No.: ( ) - _____ Call taken by operator: _____

Ticket No. Assigned to Location Request: [ ]

Co. ID # (*if assigned by Utility Service Protection Center*): _____

Person Calling _____ Company Phone: ( ) - _____

Alternate Contact Name: _____ Alternate Contact Phone: ( ) - _____

Company Address: _____

Assigned Start Date: _____ / _____ / _____ a.m. (8:00 – 12:00) / p.m. (12:00 – 5:00)

Type of Work: _____ Use of Explosives: Y / N

Potential Boring Locations Identified with White Paint and/or Stakes: Y / N / Not Applicable

Work Being Done For: (*Company or Individual Name*): _____

State: _____ County: _____ City/Place: _____

Street: _____ *(Only one street per ticket)* Zip Code: _____

Nearest Intersecting Street: _____

Map Page: _____ Grid(s): _____

Description of the area to be marked *(Provide the following: Street working on, which side of street, how far in which direction from nearest intersecting street; etc.)*:

_____

_____

_____

Map of the area to be marked faxed to the Utility Service Protection Center: Y / N / Not Accepted

Utility Service Protection Center Fax No.: ( ) - _____

CALL BACK INFORMATION RECORD

	Gas	Tele.	Elec.	Wtr.	Swr.	Cable TV	Other
Date Notified							
Time Notified							
Notified By							
Phone Number							
Marks Complete							
No Conflict							
No Facilities							

Standard Operating Procedure – 29

Aquifer Pumping Tests

Scope:

This standard operating procedure (SOP) describes the methods and techniques used to perform aquifer pumping tests on partially- or fully-penetrating test wells.

Objective:

Aquifer tests are generally performed to measure the hydraulic characteristics of a water bearing zone that has been identified and partially characterized (e.g., total depth of the water-bearing unit, groundwater flow direction, texture of the matrix, etc.) by investigations at the site. The tests generally include four separate phases: a background monitoring phase; a stepped-rate pumping test; a constant-rate pumping test; and a recovery test. Aquifer tests typically include a pumping well where the aquifer is stressed by removing formation water, and at least one observation well where the response to pumping is measured.

This SOP is designed to provide the user with a general outline for conducting each phase of the aquifer test and assumes the user is familiar with basic field procedures, such as recording field notes (SOP 1), well gauging (SOP 3) and equipment decontamination (SOPs 16 through 19). This SOP does not cover aquifer test planning (e.g., layout of the pumping and observation wells, estimation of pumping rates, selection of pump types, etc.) nor does it cover the analysis of the aquifer test results for determining the characteristics of the water-bearing zone. These topics require a significant amount of planning and are more appropriately addressed in the work plan or a dedicated aquifer test plan.

These procedures are oriented towards In situ-brand transducers and data loggers, which are the current state-of-the-art and the brand most often used by WSP. While some specifics may be slightly different for other brands of monitoring equipment, the overall sequence of work should be similar and readily adaptable to any equipment set-up.

Because of the inherent complexity and the number of different tests involved, this SOP is divided into four sections. Each section provides an overview of the objective of the test and a brief description of the methods to be used. The set-up procedures are cumulative; each phase of the aquifer test uses equipment or procedures that were used in the previous phase of the test. The reader is encouraged to read the entire SOP before beginning any onsite activities.

Preliminary Procedures

Materials:
Field notebook

Laptop computer with appropriate ports and adapters

Vented pressure transducer with internal or external data loggers

Barometric pressure monitoring equipment

Electronic water level meter or oil/water interface probe (if necessary)

Photoionization detector (PID)

Groundwater extraction pump and appropriate tubing

Power supply

Water storage tank

Flow meter(s) and/or flow gauge(s)

Stopwatch

Small (1 to 5 gallon) container with known capacity

Nitrile gloves

Heat resistant gloves

Tape measure

Exacto, pocket knife, or scissors

Pump lift line (polypropylene rope or other suitable material)

Duct tape

Indelible marker or metal file

Paper towels

5-gallon bucket(s)

Distilled water (decontamination of downhole equipment)

Plastic sheeting

Waste labels (if necessary)

1. Review site Health and Safety Plan (HASP) before mobilizing to the field. Discuss groundwater contamination constituents and specific HASP issues with Project Manager and determine the proper level of PPE.
2. Prepare field book (SOP No. 1) daily with site description, weather conditions, participants, and other relevant observations, including all data necessary to complete the hydraulic conductivity tests. Pay careful attention to the weather conditions (e.g., changes in temperature, precipitation, wind, etc.) both locally and regionally as these can adversely affect the data.
3. Survey the ambient (background) air around the base of the well casing and wellhead for organic vapors (if appropriate) using a PID, or equivalent. Record air monitoring measurements in the field book.
4. Inspect extraction well and observation/test wells (e.g., monitoring wells or piezometers) for soundness of protective casing, surface ground seal, and locking mechanism; record findings in the field book.
5. Unlock protective casing or flush mounted curb box. Remove well cap or plug, place PID probe in wellhead, and record PID response in field book. Survey breathing zone to ensure that PPE level is appropriate.
6. Place plastic sheeting around the head of the extraction well or observation well before initiating any down well measurements or testing activities.
7. Allow static water level to equilibrate, as appropriate, before gauging the wells.

8. Measure the inside diameter (ID) of the casing and record in inches. From the top of the casing, measure the depth to water (DTW) and total depth (TD) of the well (in hundredths of a foot with an electronic water level indicator and record in the field book). Water levels should be measured from the surveyor's mark at the top of the casing, or if no mark is present, from the north side of the casing. Be sure to mark a location with a metal file or indelible marker on the north side of the casing if the surveyor mark is absent and record in the field book for future reference.
9. Thoroughly decontaminate (SOP No. 17) the water level indicator cable during retrieval using distilled water and paper towels.
10. Do not create an airtight seal by replacing the well caps at the locations that will be used during the pumping tests. It is important that they be open to the atmosphere. Take steps, if necessary, to protect the openings by loosely covering the well opening with the protective steel cover or other material to prevent rain, snow, or debris from entering the well during the test.

Background Monitoring Test

This portion of the aquifer test involves monitoring each of the wells (pumping and observation wells) selected for the aquifer test for a period of time, usually a minimum of 24 hours, to identify any naturally-occurring (e.g., changes in barometric pressure, earth tides, rain events, etc.) or man-made (e.g., artificial recharge, nearby pumping wells, trains, etc.) forces that might systematically alter or potentially mask water level changes during the pumping portions of the aquifer test. Data collected from the background monitoring phase establishes a baseline that can be used to correct the pumping test data by analytically removing the non-pumping trends. Background data can also be used to help determine if the water-bearing unit is confined or unconfined.

The physical placement of the downhole equipment for this portion of the test will be used for the remaining phases of the aquifer test.

1. Assemble all of the monitoring gear (transducers, data loggers, cables, etc.). Verify the transducers have power (for transducers with onboard loggers) and sufficient cable length to reach the intended monitoring zone. Each cable should be fitted with a desiccant cartridge (provided by equipment supplier) to prevent moisture from building up in the transducer vent line. Also, verify that the transducers are rated for the intended water depths; Table 1 provides the criteria for selecting the appropriate transducer. Transducers with the lowest pressure rating appropriate for the anticipated depths should be selected as the device accuracy decreases as the pressure rating increases.

Table 1

Maximum Depth Below Water Table for Pressure Transducer (a)

Pressure Transducer (PSI)	Depth Below Water Table (feet)
10	23
20	46
30	69
50	115

a/ PSI=pounds per square inch

2. Mark each transducer (on the sonde itself) with the well identification number to avoid confusion during data recovery after all phases of the aquifer test have been completed.
3. Familiarize yourself with the data logging software. Transducers with onboard data loggers usually require an interface with a laptop or a hand-held computer running the appropriate proprietary software. Additional cables or adapters (e.g., RS-232 [serial] to Universal Serial Bus [USB]) may be necessary to physically link the computer to the data loggers.
4. Thoroughly decontaminate the pressure transducers using a non-phosphate detergent and distilled water before placement of equipment into the extraction well or observation wells. Decontaminated equipment should be placed on or wrapped in clean plastic sheeting until inserted into the monitoring well or piezometer.
5. Determine the depth below ground surface at which you will be setting the individual transducers for the pumping and observations wells:
    a. The most critical placement will be the transducer installed in the pumping well. This transducer is typically positioned below the pump (which will be installed in the next phase of the aquifer test) to allow water level measurements during periods of significant drawdown. If the well is shallow enough and the depth rating of the transducer is sufficient to withstand the pressure (Table 1), the transducer can simply be lowered to a depth just above the bottom of the well; take care to not allow the transducer to contact the bottom of the well during installation. If the transducer is to be positioned below the pump and above the bottom of the well, measure the length from the transducer bottom up the cable and mark the cable for easy reference when lowering into the well. In either case, be sure the transducer position is such that the bottom of the pump will not contact the top of the transducer.

    b. The placement of transducers in the observation wells is less critical. The units need only be positioned below the level of expected drawdown in each well (you should have some idea of this based on your site characterization and aquifer test design). As with the pumping well transducer placement, the easiest positioning is to place the transducer at a depth just above the bottom of the observation well (see Table 1).
6. Practice defining, running, stopping, and downloading data using the laptop or hand-held computer before installing any of the transducers in the pumping or observation wells. Be sure that the internal time settings in the transducers, loggers, barometric pressure monitoring equipment, laptop, and/or hand held computer are synchronized before programming any tests. This step is critical to ensuring a simultaneous start (and stop) of the test, contemporaneous data collection, and synchronized data logs.
7. Follow manufacturer's specifications to prepare the barometric monitoring equipment. The unit should be placed in the same general area as the pumping well.
8. Program the background monitoring test into each data logger and the barometric pressure monitoring equipment. The background monitoring data is recorded on a linear time scale (e.g., once every 15 minutes). As barometric pressure, tides, and other natural forces typically yield changes in the wells that occur over the space of 1 to 2 hours, data collected on a schedule of once every 15 to 30 minutes should provide a data set that is sufficiently dense to follow any systematic trends. Water levels that can change on more rapid scales (e.g., karst terrain) may require shorter sampling intervals. Check the aquifer test plan to verify the appropriate timing.

9. Secure the transducer cable to the wellhead, protective casing, or curb box using duct tape, "S-hooks", or other appropriate methods. The transducers should be secured so that they do not move vertically within the water column during the aquifer tests. Be careful not to pinch or kink the cable, which could block the transducer's vent to the atmosphere.
10. Allow the groundwater elevation in the test wells to re-equilibrate with the surrounding formation after the insertion of the transducers. The amount of time required for this step will be dictated by the characteristics of the formation. Wells with recovery times of less than a minute should be allowed to stand a minimum of fifteen minutes before initiating the background test.
11. Run the background monitoring test for a minimum of 24 hours. Do not disturb the pumping or observation wells during the monitoring period. Be sure to record any changes in the weather, especially rain or snow events, and any other environmental or physical changes (e.g., tides, train schedules, etc.) that may potentially alter the water levels in the wells. Aquifer tests conducted in karst terrain should also note regional precipitation events (i.e., within a 20 mile radius) because even distant meteoric water can quickly be transmitted over long distances through infiltration in karstic terrain.
12. Do not stop the barometric data monitoring equipment at the end of the background monitoring test. Barometric data should be collected for all phases of the aquifer test.
13. Download the groundwater and barometric pressure data to the laptop or hand-held device and create a back-up copy of the data. It is not necessary to erase the data loggers, as most contain sufficient memory to store millions of data points; leaving data on the loggers provides a secondary back-up system. Verify that all of the data has been transferred to the laptop or another storage media (e.g., compact discs) before erasing the data files from the loggers.

Stepped-Rate Pumping Test

The stepped-rate, or variable-rate, pumping test is designed to stress the aquifer with a series of short-term pumping periods with incrementally higher pumping rates to determine the maximum sustainable pumping rate. The primary purpose of the stepped rate test is to establish the pumping parameters for a constant-rate test; however, data from the stepped-rate tests can be used, if sufficient data is collected, as support for the constant-rate analysis. The stepped-rate test usually involves a minimum of three pumping periods or "steps" to establish the response of the aquifer.

The following procedures assume that the background monitoring test has been completed and the monitoring equipment remains in place.

1. The groundwater extraction method should be selected based on the depth to water, the anticipated flow rate, and the availability of power. Submersible pumps should incorporate a backflow check-valve(s) that prevents water within the riser pipe from flowing back into the well when the pump is shut off. Check the aquifer test design to verify the pump requirements and appropriate type of tubing.
2. Before installation, measure the length to the pumping intake depth specified in the aquifer test plan; the length includes the lift line and attached submersible pump or the length of tubing for non-submersible pumps (e.g., peristaltic pumps). Accurate measurements are critical to avoid contact with the transducers during installation and when using electric submersible pumps in low yield aquifers. Dry pumping electric submersible pumps, even for short periods of time, may damage the device.

3. Install and secure the tubing or submersible pump and tubing in the pumping well to the specified depth.
4. Attach a flow through meter to the discharge/conveyance line. Digital and optical styles are available, depending upon the predicted flow rates. Be sure to check the manufacturer's recommendations for calibration and ensure that the meter is equipped with the properly sized fittings. The flow meter measurements should be periodically verified by manually estimating the time necessary to fill a container of known capacity.
5. Extracted groundwater exiting the flow meter should be piped to a water storage tank with sufficient capacity. The tank should be constructed of materials that are compatible with contaminants that may be present in the groundwater and any applicable state or federal regulations (i.e., Department of Transportation), especially if the material will be managed as investigation-derived waste (SOP 26).
6. Define the stepped-rate tests in each data logger and the barometric pressure monitoring equipment. Be sure that the internal time settings in the transducers, loggers, barometric pressure monitoring equipment, laptop, and/or hand-held computer are synchronized before programming any tests. The stepped-rate test is a logarithmic test. Data is collected rapidly in the first portion of the test, often several times per second, with the time between successive water level measurements becoming progressively longer as the test proceeds. This approach yields dense data in the early portion of the test where changes in water levels are occurring rapidly and less data as the water levels approach a steady-state. Most data loggers will convert the logarithmic measurement schedule to a linear schedule at a pre-designated time, which can be programmed during the test set-up. Typical log-linear conversion times range from 1 to 5 minutes for the short-term (i.e., one hour or less) steps; however, the actual conversion time should be estimated based on the aquifer test design and adjusted (for the constant-rate pump phase to follow) in the field based on observations during the individual stepped-rate tests. Table 2 provides typical log-linear conversion times for various test durations.

Table 2

Maximum Log-linear Conversion Times for Various Pumping Test Durations

Duration of Testing (Minutes)	Maximum Log-linear Conversion Time (Minutes)
10-15	1
15-60	5
60-300	30
300-1440	60

1. Gauge the pumping and observation wells prior to beginning the tests to establish the groundwater elevation at static (i.e., non-pumping) conditions (need to ensure that the pumping well has re-established static conditions following insertion of the pump). These data will be used to determine the amount of drawdown during the tests. Wells should be gauged manually and using the transducers. Record the data in the field notebook.
2. Variable-rate tests typically consist of at least three pumping sessions with different pumping rates, one of which is the anticipated maximum extraction rate estimated from the existing site characterization data. Pumping strategies often begin at some fraction of the maximum anticipated rate (i.e., one-half) with increasing steps towards the maximum. An alternate strategy

begins with the anticipated maximum pumping rate and adjusts the steps up or down based on the drawdown observed in the pumping well. In either case, it is important that at least one of the pumping sessions be conducted at a rate that is greater than the initial estimate to verify that the maximum sustainable pumping rate has been achieved.

3. Begin the variable-rate test sequence. Careful synchronization of the start of the test and the pump are critical to ensure optimal data capture during the initial drawdown phase. Begin pumping the well 0.5 to 5 seconds after the data collection has started; starting pumping before the data loggers begin collecting data will result in the loss of important early drawdown data.
4. Monitor the pumping rate and groundwater elevation frequently during the test. Pumping rates should be maintained within 5 percent of the target extraction rate. Groundwater elevations in the pumping well should be monitored regularly using both the transducer and an electronic water level meter to ensure that the well is not dewatered during the test. Record discharge rates, meter readings, and manual water level measurements in the field book.
5. Continue each step for the prescribed amount of time, or until the water level in the extraction and observation wells reach stabilization. It is important to run the initial step long enough to establish that the effects of well storage (i.e., water stored in the well casing and surrounding filter pack) have dissipated and formation water is being drawn into the well. If the water levels do not stabilize and the continued operation of the test will expose an electric submersible pump, discontinue the test and reevaluate the pumping rate.
6. Water levels should be allowed to recover to static conditions (i.e., within 95 percent of the initial elevations) between each step. Do not remove the transducers or the pump from the test wells at the end of the test.
7. Download the groundwater elevation and barometric pressure data to the laptop or hand-held device and create a back-up copy of the data. It is not necessary to erase the data from the loggers as most contain sufficient memory to store millions of data points; leaving data on the loggers also provides a secondary back-up system. Verify that all of the data has been transferred to the laptop or another storage media (e.g., compact discs) before erasing the data files from the loggers.

Constant-Rate Pumping Test/Recovery Test

The constant-rate pumping phase of the aquifer test is designed to stress the water bearing zone over an extended period of time to evaluate the hydraulic conductivity and storage parameters of the unit. Typical constant-rate tests are run for 48 to 72 hours using the maximum pumping rate established during stepped-rate tests. The constant-rate test includes a recovery phase which occurs immediately after the pump is turned off. The recovery test is a passive test performed to monitor the response of the water-bearing zone as it recovers from the stress induced by the constant-rate test. Recovery is typically measured for 24 hours.

1. The set-up for the constant-rate test is identical to that of the stepped-rate tests. Groundwater levels should be at static, pre-pumping conditions before starting the constant-rate test.
2. Program the constant-rate test into the loggers and barometric pressure monitoring equipment. Be sure that the internal time settings in the transducers, loggers, barometric pressure monitoring equipment, laptop, and/or hand-held computer are synchronized before programming any tests. The constant rate test is a logarithmic test. Caution should be used when setting the conversion

time for the test. High sampling frequencies that are important in the early phase of pumping become less desirable as the test proceeds and the water levels stabilize. Typical constant-rate tests performed in low to moderate yield unconsolidated units have conversion times that range between 15 and 60 minutes.

3. Begin the constant-rate test. Careful synchronization of the start of the test and the pump are critical to ensure optimal data capture during the initial drawdown phase. Begin pumping the well 0.5 to 5 seconds after the data collection has started; starting pumping before the data loggers begin collecting data will result in the loss of important early drawdown data.
4. Monitor the pumping rate and groundwater elevations periodically during the test. As with the stepped-rate test, pumping rates should be maintained with ± 5 percent of the target extraction rate and the water levels above the pump should be checked to ensure that the well is not being dewatered. Groundwater elevations in the pumping well should be monitored regularly using both the transducer and an electronic water level meter to ensure that the well is not dewatered during the test. Record discharge rates, meter readings, and manual water level measurements in the field book. Monitoring should be more frequent at the beginning of the test with reductions in the monitoring schedule, as appropriate. Once stabilization has been achieved, the monitoring schedule can be reduced further; however, monitoring should be conducted, at a minimum, every two hours after the water levels have stabilized to verify that the pumping rate remains constant, the water levels have not decreased due to changes in the water-bearing unit storage, and other equipment is operating as designed. Record the monitoring intervals and results in the field book.
5. Using the data recorded by the data loggers and barometric monitoring equipment, continue the constant-rate test for the prescribed amount of time or until the groundwater level in the pumping and observation wells reach stabilization.
6. At the conclusion of the constant-rate pumping test, suspend all data recording but do not shut off the pump or remove any equipment from the wells. Download the constant-rate test data from the data loggers.
7. Program a recovery test into each transducer using a laptop or hand-held device. Be sure that the internal time settings in the transducers, loggers, barometric pressure monitoring equipment, laptop, and/or hand-held computer are synchronized before programming any tests. The recovery phase is a logarithmic test. Log-linear conversion times should be the same for the recovery period as those used for the constant-rate pumping test.
8. Begin the start sequence for the recovery test. Careful synchronization of the start of the data loggers and the pump shutdown is critical for capturing the early response data that occurs as soon as the pumps are shut down (this is the inverse of the drawdown data collected during the pumping phases of the test). The pump should be shut down approximately 0.5 to 5 seconds after the data loggers begin recording the test.
9. Continue the recovery test for the prescribed amount of time or until the water level in the extraction and observation wells has returned to 95 percent of the pre pumping static water level. Do not remove the downhole equipment (e.g., pump, transducers) until the completion of the recovery test.
10. Download the groundwater elevation and barometric pressure data to the hand-held device and create a back-up copy of the data on a laptop computer at the conclusion of the recovery test. It is not necessary to erase the data from the loggers as most contain sufficient memory to store millions of data points; leaving data on the loggers also provides a secondary back-up system.

Verify that all of the data has been transferred to the laptop or other storage media (e.g., compact discs) before erasing the data files from the data loggers.

11. Remove the pump, tubing, piping, transducer cables, and transducers from the test wells and place them on plastic sheeting around the well or in a bucket. Replace well cap, locking expandable plug, and secure test wells with locking mechanism.
12. Decontaminate transducers, transducer cables, and pump with distilled water and paper towels (SOP No. 17). Decontaminated equipment should be placed on, in, or wrapped in plastic until further use. Properly dispose of decontamination water, PPE, lift line, and other disposable supplies in accordance with the IDW plan.

Standard Operating Procedures – 30

In Situ Single Well Hydraulic Conductivity Test (Slug Test)

Scope:
This standard operating procedure describes the methods and techniques to be employed for performing in situ single well hydraulic conductivity tests, or slug tests, on partially- or fully-penetrating monitoring wells or piezometers.

Objective:
The objective of performing in situ single well hydraulic conductivity tests, or slug tests, is to measure the hydraulic characteristics of the aquifer (water-bearing zone) in the immediate vicinity of the monitoring well or piezometer for aquifer characterization. The hydraulic conductivity test is performed by creating an instantaneous change in the water level by either inserting (falling head) or withdrawing (rising head) a slug from a monitoring well or piezometer that is at equilibrium.

Materials:
Field book
Monitoring well lock keys
Hand tools (wrenches), if necessary
Water level indicator
Pressure transducer-internal/external logger (Troll, PDA, or Hermit, see Table 1)
Oil/water interface probe, if necessary
Photoionization detector (PID), if necessary
Closed volume slug (polyvinyl chloride [PVC] pipe filled with sand, cement, or other inert material)
Nitrile or heat resistant gloves
Tape measure
Exacto, pocket knife, or scissors
Rope
Duct tape
Indelible marker or metal file
Paper towels
5-gallon bucket(s)
Distilled water (decontamination of downhole equipment)
Plastic sheeting

Procedures:

1. Review site Health and Safety Plan (HASP) before mobilizing to the field. Discuss groundwater contamination constituents and specific HASP issues with Project Manager.
2. Prepare field book (SOP No. 1) with site description, weather conditions, participants, and other relevant observations, including all data necessary to complete the hydraulic conductivity tests, or slug tests. Verify monitoring well or piezometer locations.
3. With field personnel in Level D personal protective equipment (PPE), unless historical data warrants upgrading to Level C PPE, survey ambient (background) air around the base of the well casing and wellhead for organic vapors using a photoionization detector (PID) or equivalent. Record air monitoring measurements in field book.

4. Inspect monitoring well or piezometer for soundness of protective casing, surface ground seal, and locking mechanism.
5. Unlock protective casing or flush mounted curb box. Remove well cap or plug, place PID probe at wellhead, and record PID response in field book. Survey breathing zone to ensure that PPE level is appropriate. Record air monitoring measurements in the field book.
6. Place plastic sheeting around the monitoring well or piezometer head before initiating any down well measurement or testing activities. After plastic is placed around the monitoring well covering the ground surface, measurement and testing procedures may commence. If necessary, place tape on well casing to prevent cutting or slicing of rope or cable.
7. Allow static water level to equilibrate, as appropriate, before measuring or determining the presence or thickness of a floating product layer (LNAPL) or a sinking free product layerc (DNAPL). Measure thickness using an oil/water interface probe, if necessary, in accordance with EPA RCRA Groundwater Monitoring Technical Enforcement Guidance Document [November 1992], state guidance, or site work plan documents (SOP No. 3a). Record observations in the field book. If LNAPL or DNAPL are present, do NOT perform hydraulic conductivity tests in the monitoring well/piezometer.
8. Measure the monitoring well or piezometer casing inside diameter (ID) and record in inches. From the top of the casing, measure the depth (in hundredths of a foot) to water (DTW) with an electronic water level indicator and record in the field book. Static water level measurements must be recorded from the surveyor's mark at the top of the casing, if present. If no mark is present, mark a location with a metal file or indelible marker on the north side of the casing and record in the field book for future reference. Measure and record the total depth (TD), in hundredths of a foot, of the monitoring well or piezometer.
9. Thoroughly decontaminate (SOP No. 17) the water level indicator cable during retrieval from the monitoring well or piezometer using distilled water and paper towels.
10. Thoroughly decontaminate the pressure transducer and closed volume slug using distilled water before placement of equipment into the monitoring well or piezometer. Decontaminated equipment should be placed on or wrapped in clean plastic sheeting until inserted into the monitoring well or piezometer. Record pertinent data logger and pressure transducer information (model or serial number and pressure transducer rating) in field book.
11. Using the tape measure, measure the length from the transducer bottom up the cable to a point where the transducer will be positioned in the monitoring well or piezometer. Mark the transducer cable at the appropriate length for reference. The transducer must be positioned at a depth below the static water level so upon total insertion of the slug into the water column (slug completely enters the water column), the slug bottom does not contact the top of the transducer.
12. Position the transducer in the monitoring well or piezometer below the static water level to the point where mark is aligned with the monitoring well or piezometer survey mark. Allow transducer to equilibrate for a minimum of 10 minutes before starting test.
13. Secure transducer cable to the wellhead, the well casing, protective casing (i.e., duct tape), or the curb box (i.e., "s" hooks). Note, the transducer must be secured such that the transducer can NOT move vertically within the water column during performance of the hydraulic conductivity tests. If using an external data logger, connect the transducer cable to the automatic data logger.

14. Power the data logger/transducer and check for proper operation and battery life. Enter monitoring well/piezometer information, transducer reference data, and testing parameters. Generate a test file for each planned individual slug test; the number of tests to be performed should be determined by the Project Manager, but should be no less than three (two falling and one rising) per monitoring well/piezometer. Verify that data logger/transducer collection mode is programmed to record on the logarithmic scale, with a maximum asymptotic interval of 10 minutes.
15. Evaluate water level stabilization by reviewing pressure head readings from the transducer. Once pressure readings have stabilized, record the transducer reference point (zero or reference head).
16. Verify that the slug is full and closed, as applicable. Closed volume slug should be of an appropriate diameter and length to displace an appropriate water volume on insertion into the water column. Secure rope to the top of the closed volume slug; checking knot to be certain rope will not untie during testing process. Using the tape measure, measure the length from the bottom of the closed volume slug up the rope to a point where the slug will be positioned immediately above the recorded static water level. Mark this location.
17. On the rope, mark a second depth measurement corresponding to the depth at which the slug should be lowered into the water column (falling head). The slug must be positioned below the static water level upon total insertion into the water column, but above the level of the transducer. Secure the closed volume slug rope to well head, the well casing, protective casing, or the curb box using duct tape or some other means.
18. In preparation for the first test, lower the closed volume slug into the monitoring well or piezometer so that the slug bottom is suspended immediately above the static water column. This position should correspond to the first mark (No. 16 above).
19. Falling-Head Tests
    Start the test on the data logger and, approximately 0.5 second to 1 second later, smoothly but quickly lower the closed volume slug into the water column (falling- head test, slug in) until the second mark is positioned equal to the wellhead survey mark. Be sure that the slug does not move the transducer cable.
20. Rising-Head Tests
21. Start the data logger and approximately 0.5 second to 1 second later, smoothly, but quickly, remove the closed volume slug from the water column (rising-head or bail-down test, slug out) until the first mark is positioned equal to the wellhead survey mark (ensuring that the slug is above the original static water level). Be sure that the slug does not move the transducer cable. 20. During each hydraulic conductivity test, monitor (review) the water level data collected on the data logger to ensure proper operation. Record weather changes, heavy equipment movement, or other conditions that may affect water level measurements in the field book. In addition, verification should also be conducted to determine that the volume of the slug is roughly equal to the volume of water displaced, as measured by the data logger. Using the data recorded by the data logger, continue the hydraulic conductivity test, or slug test, until the water level in the monitoring well or piezometer returns to the smaller of either 0.02-foot from the original static water level or within five percent of the change in head.
22. Using the data logger, stop collecting measurements when the static water level stabilizes as described in Step 20.

23. After completion of the testing procedures at a location, remove closed volume slug, transducer cable, and transducer from the monitoring well or piezometer and place on plastic sheeting around the well or in a bucket. Replace well cap, expandable plug, curb box top and secure monitoring well or piezometer with locking mechanism.
24. Decontaminate transducer, transducer cable, and closed volume slug with distilled water and paper towels (SOP No. 17). Decontaminated equipment should be placed on, in, or wrapped in plastic until further use. Properly dispose of decontamination water, PPE, rope, and other disposable supplies in accordance with the investigation-derived waste (IDW) plan.
25. In the field, periodically, and/or before concluding the field activities, download data files from the transducer/logger to a computer diskette or laptop computer.

General Notes:
If groundwater quality data is available for the well(s) being tested, conduct the slug tests in the order of increasing contaminant levels to reduce the potential for cross-contamination.
Single well hydraulic conductivity test, or slug test, data should be analyzed using appropriate aquifer type (unconfined, confined, etc.), method (Bouwer and Rice, Hvorslev, etc.), monitoring well or piezometer construction parameters (fully- or partially-penetrating), pre-testing water level measurements, and HydroSOLV's AQTESOLV for Windows (or equivalent) software package.
Hydraulic conductivity (K) values are calculated (length/time) as centimeters per second (cm/sec), meters per second (m/sec), or feet per second (ft/sec).

Pressure transducer selection criteria:

Table 1
Maximum Depth Below Water Level for Pressure Transducer (a)

Pressure Transducer (PSI)	Depth Below Water Table (feet)
11	23
21	46
31	69
51	115

a/ PSI=pounds per square inch

Standard Operating Procedure - 31

Screening of Soils for Metals Using a Delta Handheld X-Ray Fluorescence Analyzer

Materials:

Personal protective equipment (PPE)
Sample containers
Sample labels and indelible marker
Stainless steel trowels or spoons
Field log book
Delta XRF
Battery pack and charger
Shielded case
Calibration standards
Test platform
Ziploc bags
Test guard
Baby wipes
Sample tray

Note: Prior to obtaining a rental XRF unit, determine the requirements for radioactive material licensing and training from the State's Radiological Materials Department.

Calibration:

1. Every time the XRF is turned on or reset, the instrument performs a self-calibration. In addition, several known standards are provided to check the calibration of the instrument. Calibrate the XRF at least daily, preferably each morning prior to conducting soil screenings, in the middle of the day, and at the end of day following soil screenings. Calibrate the XRF in accordance with the manufacturer's specifications using the low, medium, and high calibration standards.

2. Allow the instrument 15 minutes to warm up prior to field use.

In-situ Field Screening:

1. Select a measurement site. Always use the appropriate personal protective equipment based on the site conditions.

2. Clear any surface debris or vegetation from the measurement location, and ensure a flat surface to the soil. If necessary level the surface with a stainless-steel trowel or spoon.

3. The surface will be tamped to increase soil density, based on the SW-846 protocol; this process will allow better repeatability and ensure that the sample is representative.

4. Place the test guard on the ground.

5. Hold the Delta in one hand, and push the safety slide (that locks the shutter release) out from under the shutter release.

6. Place the Delta on the test guard so that the rectangular opening on the test guard is under the window of the Delta. Squeeze the shutter release and firmly press the instrument flat against the surface of the test guard (if you don't squeeze the shutter release, the plunger will not depress and the shutter will not fully open causing an inaccurate measurement). **Do not put your hand on the end plate of the unit while measuring. Never point the unit at yourself or anyone else when the shutter is open.**

7. Watch for indications to decide when the test has reached the desired level of accuracy. A typical screening will last 30-60 seconds.

7. The instrument will provide a reading in ppm plus a measurement precision (+/-). Add the precision to the reading of concern to determine the metal concentration at that location.

8. Clean the bottom (soil contact side) of the Delta Test Guard with a baby wipe prior to the next screening location.

Note: In the unlikely event that the plunger gets stuck in the open position, simply push it closed. Then call the Delta Service Department at (866) 446-6689.

<u>*Ex-situ* Field Screening:</u>

1. Using the trowel loosen and homogenize the soil to the desired depth.

2. Place 50-100 grams of soil in a new plastic bag and close securely.

3. Label each bag with the sample location/ID.

4. Break down large clumps of soil within the plastic bag while further homogenizing the sample.

5. Shape the bag of soil to form a continuous uniform layer of at least one centimeter thickness and analyze the sample using the *in-situ* screening procedure (steps 3-8).

Note: Test results will tend to be 10-15% lower than results obtained using the *in situ* screening procedure.

<u>Soil Moisture Content Screening:</u>

The XRF device does not perform well for saturated soils. Therefore, WSP will take the following precautions during XRF sampling:

1. WSP will not conduct XRF sampling unless there has been no precipitation at the site for at least 48 hours.
2. WSP will not conduct XRF sampling during a storm event.
3. WSP field representatives will ensure soil is not saturated prior to XRF analysis by conducting soil moisture measurements at each sampling location using a soil moisture meter (e.g., General DSMM500 Soil Moisture Meter).

4. If soil moisture content is greater than 20%, a new location within the sampling grid will be selected and the soil moisture content will be measured. This procedure will continue until a location within the grid is identified as having a soil moisture content of less than 20%.
5. If a viable sampling location is not available within the selected grid, a soil sample from within the grid will be collected in accordance with WSP SOP #2 for fixed-laboratory analysis.

Standard Operating Procedure - 32
Composite Soil Sampling

Application:

As with soil sampling, composite samples can be collected at the surface or at depth, and different equipment is required in each instance. Surface samples can be collected most efficiently with a trowel or scoop. For samples at depth, a decontaminated, clean bucket auger may be required. For a sample core, waste pile samplers may be used. To collect representative samples of a waste pile, composite samples may be required. The objectives of the sampling program will dictate the need to collect composite samples. A grid system set-up on the waste pile may also be used to collect individual or composite samples.

Materials:

Stainless steel trowels or scoops

Stainless steel spoons

Bucket augers

Extension rods

Auger handle

Pipe wrenches

Teflon tape

Work, nitrile, and surgical gloves

Saranex or Tyvek suit

Eye protection

Respirator (if necessary)

Analytical buckets

Tray, mixing pans

Ziploc® plastic bags

Stakes and string (grid system)

Procedure:

1. Soil samples collected from bucket augers, stainless steel spoons, trowels or scoops, will be placed in plastic bags to be homogenized with the additional samples intended for compositing. Each bag of soil shall be transferred to a clean tray or mixing pan for compositing. Use a clean, decontaminated spoon, scoop, or trowel to homogenize the sample. Remove any foreign matter (e.g., rocks, sticks, or other debris) from the tray. Transfer the homogenized sample directly into a pre-labeled sample container. The laboratory should be contacted in advance so they can provide the appropriate sample containers for the analysis required. A wide-mouth sampling

container is preferable for containing the sample, as it requires less disturbance of the waste sample as the sample is being transferred into the labeled sample container. Enough soil will be collected in total to fill the sample container.

2. Keep detailed notes in the field log book of the sample location, details about the grid spacing, sample description, sample depth, sampler's name, and the requested analytical parameter(s).
3. Complete the chain-of-custody forms with the appropriate sampling information.

Standard Operating Procedure - 33

Interstitial Pore Water Sampling and Analysis

Materials:

Personal protective equipment (PPE)

Sample containers

Sample labels and indelible marker

Stainless steel trowels or spoons

Field log book

Sediment Sampling for Interstitial Pore Water Laboratory Analysis

Below is an explanation of interstitial pore water and recommend procedures if the scope of work requires the extraction and analysis of interstitial pore water in sediment samples.

Sediment interstitial water, or pore water, is defined as the water occupying the spaces between sediment particles. Interstitial water might occupy about 50% (or more) of the volume of a depositional (silt-clay) sediment. The interstitial water is in contact with sediment surfaces for relatively long periods of time and therefore, might become contaminated due to partitioning of the contaminants from the surrounding sediments. In addition, interstitial waters might reflect ground water – surface water transition zones in upwelling or downwelling areas. In these areas their chemistry might be more reflective of ground or surface waters at the site. Therefore, flow, residence time and other physicochemical factors (e.g., pH, temperature, redox potential, organic carbon, sulfides, carbonates, mineralogy) might have varying roles in determining whether interstitial waters are contaminated.

In many depositional sediments, interstitial waters are relatively static, and therefore contaminants in the interstitial water and in the solid phase are expected to be at thermodynamic equilibrium. This makes interstitial waters useful for assessing contaminant levels and associated toxicity. Interstitial water is often isolated to provide either a matrix for toxicity testing and/or to provide an indication of the concentration and/or partitioning of contaminants within the sediment matrix.

Interstitial water sampling is most suitable for sediment types ranging from sandy to uncompacted silt-clays. Such sampling is not typically performed on sediments with coarse particle size (such as gravel) or on hard, compacted clays, as the potential for interstitial water contamination in the sediment types is relatively low.

When field indicator parameters require pH measurements for sediment, below is a brief discussion on procedures to follow.

Field Measurements for PH

pH is a measure of the effective concentration (or activity) of hydrogen ions and is expressed as the negative base-10 logarithm of the hydrogen-ion activity in moles per liter. Uncontaminated groundwater typically exhibits a pH ranging from 5 to 9 Standard Units (SU). Changes in pH from background may indicate the presence of groundwater contamination or that existing contamination has spread and can be very useful in identifying well construction or maintenance problems.

After calibrating the monitoring equipment, follow the manufacturer's instructions to select the display for readings the necessary geochemical parameters. The Horiba D51 equipped with a Horiba 9615-10D or 9625-10D pH electrode should be able to penetrate soft sediments. However, penetration into clays, sands and gravel conditions may not be achievable.

The following procedures should be followed and all observations and measurements recorded in the field book.

- Collection of field parameters should be completed within 15 minutes of sample collection
- Immerse the probe in the sediment (in-situ or within the sample container) and wait for stabilization of the reading before recording the measurement.
- If collecting field measurements ex-situ, agitate or swirl the sample during the stabilization period.

Record all field-testing measurement data, to include the following:

- Project name and location
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

Ex-situ Extraction of Interstitial Water

Ex-situ interstitial water collection methods are often necessary when relatively large volumes of interstitial water are required (such as for toxicity testing), when *in-situ* collection is not viable or when a brief sampling time is critical. While these extraction methods can be done in the field or in the laboratory, extraction in the laboratory, just prior to analysis or testing, is preferable so that the sample is maintained as close to its original state as much as possible during transport and storage.

General Procedures

Centrifugation and squeezing are the two most common techniques for collecting interstitial water, and are generally preferred when large volumes are required. Other methods include

pressurization (e.g., vacuum filtration) devices, which can be used to recover small volumes of interstitial water.

Regardless of the method used, interstitial water should be preserved immediately for chemical analyses, if appropriate, or analyzed as soon as possible after sample collection if unpreserved (such as for toxicity testing). Significant chemical changes can occur even when interstitial water is stored for periods as short as 24 hours.

If sediments are anoxic, as most depositional sediments are, sample processing, including mixing of interstitial water that has separated from the sediment, should be conducted in an inert atmosphere or with minimal atmospheric contact. Exposure to air can result in oxidation of contaminants, thereby altering bioavailability. Air exposure can also result in loss of volatile sulfides, which might increase the availability of sulfide-bound metals. In addition, iron and manganese oxyhydroxides are quickly formed upon exposure to air. These compounds readily complex with trace metals, thus altering metals-related toxicity. Maintaining anoxic processing conditions is not necessary when study objectives are concerned with exposures to aerobic sediments, or if target contaminants are unaffected by oxidation in short-term toxicity or bioaccumulation testing.

Interstitial water filtration should be avoided. Numerous studies have shown that filters reduce toxicity and contaminant concentrations by retaining contaminant-associated particles and also by contaminant sorption onto the filter matrix. If filtration is stipulated by a test method, treated filters (e.g., pre-soaked in distilled, deionized water, or combusted at 400° C overnight for glass fiber filters) should be used, and an unfiltered sample should also be tested for toxicity and contaminant concentrations. The characteristics of filters and the filtering apparatus should also be carefully considered, as different filters have different sorptive capacities for different contaminants.

<u>Extraction of interstitial water</u>

- *Centrifugation is the generally preferred laboratory method for the extraction of interstitial water.*
- *Extraction of interstitial water should be completed as soon as possible.*
- *Interstitial water that has accumulated on the surface of the homogenized sediment sample should be mixed into the sediment before the sample is partitioned among centrifuge bottles.*
- *Unless site-specific information suggests otherwise, centrifuging should be at 4° C to minimize temperature-mediated biological and chemical processes.*
- *Interstitial water should be preserved immediately for chemical analyses or analyzed as soon as possible after extraction, unpreserved. For toxicity testing, interstitial water should be stored at 4° C for not longer than 24 hours, unless the test method dictates otherwise.*
- *Filtration should be avoided unless required by a test method because it might reduce interstitial water toxicity. Double (serial) centrifugation (low speed followed by high speed) should be used instead.*

Centrifugation

Centrifugation is the generally preferred laboratory method for collection of interstitial water. It is a relatively simple procedure that allows rapid collection of large volumes of interstitial water. It also facilitates the maintenance of anoxic conditions (if required). However, centrifugation, like other *ex-situ* procedures might yield chemical and/or toxicological artifacts due to the extraction procedures themselves, which might alter the natural equilibrium between interstitial water and sediment.

Prior to centrifugation, the sediment sample is homogenized and partitioned among centrifuge bottles. If the homogenized sample is stored prior to centrifugation, interstitial water might accumulate on the surface of the sediment. This overlying water should be mixed into the sediment before subsampling for centrifugation. Samples are then partitioned among centrifuge bottles. In general, approximately 50% of sediment moisture content can be extracted as interstitial water. If interstitial water volume requirements are lower, smaller sediment subsamples may be used.

Centrifugation has been performed at various temperatures. ASTM (2000a) recommends that the centrifugation temperature reflect the in situ sediment temperature to ensure that the equilibrium between the particulate and interstitial water is not altered. Alternatively, a temperature of 4° C may be preferred to minimize temperature-mediated chemical and biological processes.

When centrifuging coarse sand, it might be desirable to use a modified centrifuge bottle to aid interstitial water recovery. The modified bottle is equipped with an internal filter that can recover 75% of the interstitial water, as compared to 25 - 30% recovery from squeezing.

Below is an example of laboratory procedures for Centrifugation of Sediment for Pore Water collection provided by RTI Laboratories (Michigan).

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ANALYTICAL STANDARD OPERATING PROCEDURE

Centrifugation of Sediments

SOP#:	**PWCEN_022714_R0**
Method Reference:	**EPA 8272**
Issue Date:	**February 27, 2014**
Revision No.:	**0**
Revision Date:	**February 27, 2014**

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Director, Quality Management: Charles O'Bryan

Director, Environmental Services: Yemane Yohannes

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STANDARD OPERATING PROCEDURE

Centrifugation of Sediments SOP#: PWCEN_022714_R0

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1.0 Scope and Application:

1.1 Introduction

RTI Laboratories, Inc. has prepared this document to detail the Standard Operating Procedure (SOP) for the centrifugation of sediments

A detailed description of the instrumentation, calibration, run parameters and quality control procedures is included in this SOP. For definition of terms not specifically defined in this document refer to RTI QAP Section 16.

1.2 Summary of Method

1.2.1 This standard operating procedure (SOP) provides general technical requirements and operational guidelines for collecting pore water samples from sediment. The requirements of this SOP are applicable to sampling of pore water from sediment collected in dredging, streams, rivers, lakes, ponds, swamps, wetlands and drainage ditches.

2.0 Safety Precautions

2.1 Waste materials submitted for analyses may contain hazardous components. Uncharacterized waste samples should be handled in a manner that minimizes exposure and personal contact. Analysis should be performed in a fume hood.

2.2 Chemicals used should be regarded as a potential health risk. Sample preparation and analysis is performed in a manner designed to minimize exposure using routine good laboratory practices.

3.0 Sample Requirements and Sample Handling Procedures

3.1 Samples are received in accordance with RTI Laboratories, Inc. Standard Operating Procedure for Sample Receipt and Custody (SRC001-A). Appropriate U. S. Environmental Protection Agency (EPA) sample handling procedures and sample preservation recommendations are followed.

3.1.1 Samples are collected in a clean container with sufficient material to yield enough water after centrifugation to perform the requested analyses.

3.1.2 Sample holding time is 28 days from collection.

3.1.3 Holding time for the generated water sample is dependent on the analytical method being performed.

4.0 Apparatus and Materials

4.1 Centrifuge capable of >1000 g.

4.2 Centrifuge buckets capable of holding at least 1 L

4.3 Balance capable of measuring up to 5 kg to the nearest gram.

4.4 Syringe and 0.45 μm syringe filters (disposable)

4.5 Clean, disposable pipettes.

4.6 Timer (0 to 30 minutes)

4.7 Scrub brushes

5.0 Reagents

5.1 DI Water-for cleaning purposes

5.2 Laboratory soap

6.0 Sample Preparation

6.1 Divide sample by weight into each of the two centrifuge buckets (for balancing purposes) to the nearest gram.

6.2 The centrifuge should be set at 1000-1500 rpm. These settings have been determined to be adequate to generate maximum pore water from samples

6.3 The sample will be spun for up to 30 minutes; however 15 minutes is usually sufficient to achieve pore water separation from the sediment. All samples will be centrifuged for the same period of time to ensure consistency.

6.4 Once the centrifuge rotor has come to a complete stop, the buckets should be carefully removed from the swing are to avoid unsettling the sample.

6.5 Carefully pour the supernatant into pre-cleaned, labeled wide-mouth jars. Clean, disposable pipettes may be used.

6.6 Filter appropriate amount of sample using a disposable syringe and 0.45 μm disposable filter.

6.7 Store all samples, supernatants and filtrates at 4 °C.

6.8 If no liner is used for the centrifuge buckets, they must be scrupulously cleaned using DI water and laboratory soap. Additionally, before each use they must be rinsed several times with DI water and dried thoroughly.

6.9 pore water preparations blank will be processed through all of the steps in this procedures using laboratory DI water as the sample matrix.

7.0 Preventative Maintenance and Routine Cleaning

7.1 If no liner is used for the centrifuge buckets, they must be scrupulously cleaned using DI water and laboratory soap. Additionally, before each use they must be rinsed several times with DI water and dried.

8.0 Waste Management and Pollution Prevention

8.1 Pollution Prevention

8.1.1 Reagents are purchased and quantities used to reflect anticipated usage and minimize disposal of expired or unused chemicals.

8.1.2 Chemicals are handled in a manner that minimizes the potential for release or spill of the material.

8.2 Waste Management.

8.2.1 Acidic materials (pH<2) are treated as hazardous waste and placed in the appropriate waste drum for disposal.

8.2.2 Soil samples are composited in a drum designated for incineration. Disposal of materials is addressed in the laboratory QAP (Section 5.6).

9.0 References

9.1 EPA Test Methods for Evaluating Solid Waste. Laboratory Manual Physical/Chemical Methods, Method 8330B, Revision 2 October 20EPA 8272.

9.2 RTI Laboratories, Inc. Quality Assurance Plan.

9.3 RTI Laboratories, Inc. SOP SRC001-A, Sample Receipt and Custody.

9.4 RTI Laboratories, Inc. Chemical Hygiene Plan

9.5 RTI Laboratories, Inc. Employee Handbook

Standard Operating Procedure - 34

Multi-habitat (MHAB) Benthic-Macroinvertebrate Collection Procedure

Modified from Indiana Department of Environmental Management (IDEM)

Technical Standard Operating Procedure S-001-OWQ-W-BS-10-T-R0 (IDEM, 2010)

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Attachment A - IDEM Qualitative Habitat Evaluation Index (QHEI) form

1.0 Scope of Operations

The purpose of this technical standard operating procedure is to ensure that the sampling of benthic macroinvertebrate communities using the Multi-Habitat (MHAB) benthic-macroinvertebrate sampling method is completed in a consistent manner. This TSOP was modified from Indiana Department of Environmental Management (IDEM) Technical Standard Operating Procedure (S-001-OWQ-W-BS-10-T-R0). The macroinvertebrates are being collected for the purpose of analyzing the organism's tissue for Resource Conservation and Recovery Act (RCRA) metals using EPA method 6020.

2.0 Checklist of Equipment for Macroinvertebrate Collection

- Insect repellent
- IDEM Qualitative Habitat Evaluation Index (QHEI) form and maps
- Biological Samples Field Chain of Custody form

- Clipboard
- Field notebook
- Permanent marker
- Lead pencils
- D-frame dipnet (with extra net and clips) consisting of a wooden 52-inch handle attached to a 1 foot x 1 foot D-shaped frame fitted with a net with a mesh size of 500 micrometers
- Two No. 35 Sieves mesh size of 500 micrometers (0.0197 inches)
- Two plastic sample buckets, 10 quart capacity with handle and pouring spout
- White plastic sample tray (18 inches x 14 inches x 3.5 inches)
- Two pairs of forceps 4A or "fine-tip" forceps
- Chest waders
- Wading boots
- Hip boots (optional)
- Arm length rubber gloves
- Safety goggles or glasses
- Ice chest filled with wet or dry ice
- Chain of Custody forms
- Ziplock bags or other sampling containers
- 25-meter measuring tape
- Brush for cleaning sampling equipment

3.0 Definitions

"Glide" – An area common to most modified stream channels that do not have distinguishable pool, run, and riffle habitats; the current and flow is similar to that of a canal; the water surface gradient is nearly zero (Ohio EPA 2006).

"Pool" – An area of the stream with slow current velocity and a depth greater than riffle and run areas; the stream bed is often concave and stream width frequently is the greatest; the water surface slope is nearly zero (Ohio EPA 2006).

"Riffle" – The shallow parts of the stream where water flows swiftly over completely or partially submerged pebble to boulder sized rocks to produce surface agitation (Ohio EPA 2006).

"Run" – Areas of the stream that have a rapid non-turbulent flow; runs are deeper than riffles with a faster current velocity than pools and are generally located downstream from riffles where the stream narrows; the stream bed is often flat beneath a run and the water surface is not visibly broken (Ohio EPA 2006).

"X-Point": The point in the stream or pond where the sample was proposed. It should be included sampling.

4.0 Training and Experience Requirements

The field team should consist of at least two people. The person in charge of collecting the benthic invertebrate samples should have previous field experience with the collection and identification of aquatic macroinvertebrates. A familiarity with local flora and fauna (beside macroinvertebrates) in order to recognize poisonous species is highly recommended.

Sampling on surface waters requires safety consciousness of staff members. Team members should be familiar with site-specific hazards and emergency response.

5.0 Field Procedures

5.1 Stream Sampling Locations

If multiple samples are to be collected in the stream, work from the downstream-most location first and move upstream for each additional sample.

Document conditions of each sampling location in the field notebook on the Qualitative Habitat Evaluation Index (QHEI) form (Attachment A). The field notebook can reference the QHEI form and does not have to duplicate the information. Information to capture includes:

General Information

Stream Name/Location: The name of the stream and the road nearest to the stream (include GPS coordinates)

NPS Pollution: Note any potential sources of pollution in the sampling reach (litter, aboveground or below ground storage tanks, high traffic, etc)

Predominant Surrounding Land Use: Describe the predominant land use in the area surrounding the sampling reach.

Canopy Cover (% Open): Enter an estimation of the percent of stream area in the sampling reach not shaded by riparian vegetation.

Engineering: Note if the stream has been channelized or if a dam is present.

Riparian Zone/In-stream Features

Erosion: Describe any significant bank erosion in the sampling reach.

High Water Mark: If there is evidence of recent high flow (debris collected in tree limbs, traces of mud left on riparian vegetation) enter (in meters) an estimation of the maximum height of flow, above the current stream level.

Stream Depth: Enter an estimation (in meters) of the average riffle, run, and pool depths, if they were present at the site.

Stream Width: Enter (in meters) the width of the stream at the x-point.

Distances: Enter an estimation (in meters) of the distance between riffles or bends if more than one riffle or bend are present within the stream reach visible from the x-point.

Sediment

Sediment Odors: Note any odors that were detected during the sampling event.

Sediment Deposits: Check any sediment deposits that were encountered in the sampling reach.

Sediment Oils: Check one to provide an estimation of sediment oils (if any) that were encountered during the sampling event. If the undersides of un-embedded stones are black, check the appropriate box.

Substrate Components

Inorganic Substrate Components: Enter in percentages of multiples of 5 (0%, 5%, 10%, etc.) the amount of substrate composed by Bedrock, Boulders, Cobble, Gravel, Sand, Silt, or Clay.

Organic Substrate Components: Enter in percentages of multiples of 5 (0%, 5%, 10%, etc.) the amount of substrate composed by Detritus (as sticks or wood), Detritus (as CPOM), Muck/Mud, or Marl.

Water Quality

Water Odors: Check any water odors that were detected during the sampling event.

Water Surface Oils: Check any water surface oils that were detected in the sampling reach.

Stream Type: Check whether the stream was warm or cold water. Criteria for this designation can be found in Aquatic Research Center of the Indiana Biological Survey 2007.

Turbidity: Provide an estimation of the streams turbidity in the sampling reach.

Document GPS coordinates of sampling locations in field notebook and on field map. If a sample could not be collected note the reason.

Take upstream and downstream pictures of the sampling location.

Visually survey the in-stream and surrounding riparian habitat of the right and left banks of each stream reach 25 meters upstream and 25 meters downstream of the x-point. These 25-meter areas of the stream should be accurately measured using a 25-meter measuring tape or a rangefinder. Ideally, a good quality, wadeable riffle would be located within 25 meters of the x-point; if this is not the case, then a riffle located no more than 15 times the stream's wetted width (to a maximum of 500 meters) from the x-point may be sampled. If a riffle is not available, the crew leader should locate a good quality wadeable run or glide habitat to be sampled during the kick portion of the MHAB sampling procedure. A 1-minute kick sample is not collected in deep U-shaped (or bowl shaped) streams and in

streams where low-flow has reduced the stream to interstitial flow between a series of pools; in these instances the 25-meter MHAB sweep by itself is sufficient.

Identify the 25-meter section of stream shoreline habitat that will potentially yield the most diverse macroinvertebrate sample. The quality of the sample can be improved by selecting the section of stream featuring the largest number of productive in-stream macroinvertebrate habitat types, avoiding collecting samples downstream of bridges and tributaries flowing into the stream and attempting to collect the sample from an area that contains a riffle-run-pool complex. The x-point (which is fixed and must, under normal circumstances, be included in the macroinvertebrate sample) may be located at the "top" of the sampling reach (by sampling a 25-meter stretch of one bank downstream of the x-point), at the "bottom" of the sampling reach (by sampling a 25-meter stretch of one bank upstream of the x-point), or may be in the middle of the sampling reach (for instance, sampling 10 meters upstream and 15 meters downstream of the x-point on one bank). If necessary, the sampling reach may also be defined as two alternating or parallel sections of opposite banks that, combined, total 25 meters and includes the x-point.

If the actual x-point is located on a section of the stream that is currently dry or inaccessible, then the sample may be collected in a section of the stream containing water that is within 15 times the wetted width of the stream from the x-point. This adjustment to the sampling method should be used with reservation and only if the resulting sample location will provide similar habitat to what would have been found at the actual x-point. The reason for such a modification and the area of the stream sampled should be described in the field notebook and on the drawing of the site made in the QHEI form.

In the riffle kick portion of the MHAB sample locate an area in the lower (downstream) area of the riffle or run habitat that is to be sampled. While holding the d-frame net handle, the flat end of the d-frame net is placed securely onto the substrate so that there are no gaps under the frame. Disturb (by kicking) the riffle/run substrate one foot directly in front of the d-frame net so that any dislodged macroinvertebrates are carried by the current into the net. Move in an upstream or diagonal upstream direction across the riffle/run, continuously disturbing the substrate in front of the d-frame net, for a distance of about 10 feet (this is roughly equivalent to sampling one square meter of riffle/run habitat). After completion of the kick, inspect the contents of the net. Any stones (gravel-sized or smaller) or debris (leaves or small sticks) that are kicked into the net are retained. Cobble and large sticks kicked into the net should be visually inspected and any attached macroinvertebrates removed and placed in the net; the cobble or large sticks are then returned to the stream. Streams with little or no flow may require the crew leader to disturb the substrate and sweep the d-frame net through the disturbed water column above the substrate to collect this portion of the sample. After completion of the kick the contents of the d-frame net are emptied into a plastic bucket filled half full with water. This bucket should be left at the sample processing location to reduce the risk of losing the macroinvertebrate sample and may be covered to prevent the escape of any flying species (mostly adult *Coleoptera* and *Hemiptera* species).

The sweep portion of the sample begins with a team memeber at the lower (downstream) portion of the 25 meters of shoreline habitat that is to be sampled. The d-frame net is swept over the shoreline habitat so that the net dislodges any macroinvertebrates on the substrate surface and pushes them into the water column. The d-frame net is then swept through this disturbed area above the substrate two or three times to collect any dislodged macroinvertebrates. Sweeps are conducted at roughly 1.5 to 2-meter intervals for the entire length of the 25 meters of shoreline habitat for a total of about 25-30 sweeps. Sample as many microhabitats (rootwads, emergent vegetation, woody debris, leaf packs, sandy and silty substrates, and cobble and gravel substrates) as possible, sampling these microhabitats in relative proportion to their occurrence in the 25 meters of shoreline habitat. Care should be taken to avoid collecting large amounts of debris in the net as this will slow down the elutriation and picking processes.

After completion of the 25-meter habitat sweep, empty the contents of the net into the same bucket with the contents of the kick component of the sample.

Remove and visually inspect any cobble, large gravel, woody debris, and large leaves that were emptied into the bucket. During this and the following portion of the sample, the crew member should wear elbow length rubber gloves to minimize the risk of injury. Any macroinvertebrates found on these items are removed with forceps and placed into the white plastic tray. After inspection, these debris items are discarded. Due to the conservation status of many species, any freshwater mussels of the family *Unionidae* collected in the sample should be returned to a suitable area of the stream as soon as possible (their presence and species, if known, should be recorded on the stream data sheet and in the field notebook).

After removal of large items, the crew member will use their arm to swirl the contents of the bucket and then pour the water through a No. 35 sieve. The water flow from the bucket should be continuous but stopped before any sand is poured into the sieve. After each pour, the contents of the sieve are emptied into the white plastic tray. The bucket containing the sample is then refilled with stream water using a separate bucket (taking care to not add any additional macroinvertebrates via the refill bucket). This process is repeated until the water poured through the sieve is clear and free of silt. After the elutriations, the crew leader will inspect the contents of the bucket and remove any heavier organisms that were not emptied into the sieve (e.g., crayfish, snails, and Asian or fingernail clams). Handfuls of any remaining sand, leaves or fine gravel are then removed, visually inspected for any remaining organisms, and discarded.

The objectives of the pick are to collect enough organisms to meet the laboratory's minimum required sample mass (5 grams) and also if possible collect as diverse a community of organisms as possible. If known, identify the species collected in the field notes and their relative abundance. Use a pair of forceps to individually pick macroinvertebrates from the contents of the tray and deposit them into a Ziplock bag (doubled-bagged) and placed immediately on ice.

The bags should be pre-labeled with an indelible waterproof marker. Preserving the organisms for later identification of the species is not within the scope of this procedure therefore an ethanol solution will not be used. A laboratory courier will pick samples up daily and freeze them.

Fill out a sample label and insert it into the macroinvertebrate sample bags. If multiple sample containers are used for a single sample, the labels should also include "1 of 2" and "2 of 2."

5.2 Pond Sampling

Sampling for the invertebrates in a pond should be should be conducted in a similar manner to sampling in the stream except that because water is not moving rift sample techniques will not be possible. It may be necessary to disturb more of the bottom sediments to collect enough organisms. Sampling should start on the x-point and move away slowly in a spiral, expanding the size of the sample area just enough to collect the required sample mass.

6.0 Interferences

Heavy rains of more than one or two inches can often scour the stream substrate, removing many macroinvertebrates. Sampling a stream soon after a scouring event may result in a sample of poorer quality than what would be normal. A stream should not be sampled less than two weeks after a scouring event to allow for natural macroinvertebrate recolonization by drift.

7.0 Quality Assurance/Quality Control

If enough organisms are present to meet the minimum mass required for analysis; ten percent of sites sampled for macroinvertebrate community are re-sampled for QA/QC purposes. These duplicate samples are collected during the same sampling event as the original sample.

8.0 Sample Labeling and Handling

Each sample label should have the following information and should be copied in the field notebook and chain of custody form.

- Sample ID
- Sample Location
- Sampler name
- Date
- Time
- Analyses required
- Number of containers (bags)

An indelible waterproof marker can be used to write the sample information directly on the outer-most sample bag. Once labeled the samples should be place immediately in an ice chest filled with wet or dry ice. Samples should be covered in ice and not just placed on top. Samples should be double-bagged to prevent leakage. Contact the laboratory courier to pick the samples up. The courier should take the samples directly to the local laboratory where they will be checked in and placed in a freezer within 2 hours of receipt. The samples will not be allowed to thaw until they are ready to be processed by the laboratory.

9.0 Equipment Cleaning

After collecting the sample the sample tools (i.e., nets, forceps, tray, sieves, buckets) should be washed clean in the stream or pond to remove any trapped sediment. A second rinse of the equipment should be performed at the next sampling locations just prior to collecting the next sample.

10.0 References

IDEM. 2010. Multi-habitat Macroinvertebrate Collection Procedure, Technical Standard Operating Procedure S-001-OWQ-W-BS-10-T-R0, October 30, 2010

Ohio EPA. 2006. Methods for Assessing Habitat in Flowing Waters: Using the Qualitative Habitat Evaluation Index (QHEI). Ohio EPA Technical Bulletin EAS/2006-06-1. Web address as of September 16, 2010: http://www.epa.state.oh.us/portals/35/documents/QHEIManualJune2006.pdf

Sobat, S.L. 2006. Biological Studies Section Qualitative Habitat Evaluation Index (QHEI). IDEM Standard Operating Procedure S-001-OWQ-A-BS-06-S-R1. Biological Studies Section, Assessment Branch, Office of Water Management, Indiana Department of Environmental Management, Indianapolis, Indiana.

SOP 34 Attachment A

OWQ Biological Studies QHEI (Qualitative Habitat Evaluation Index)

IDEM

Sample #	bioSample #	Stream Name	Location

Surveyor	Sample Date	County	Macro Sample Type	☐ Habitat Complete	QHEI Score:

1] *SUBSTRATE* Check ONLY Two predominant substrate TYPE BOXES; estimate % and check every type present

BEST TYPES

PREDOMINANT P	R		PRESENT P	R	TOTAL %
☐	☐	BLDR/SLABS [10]	☐	☐	____
☐	☐	BOULDER [9]	☐	☐	____
☐	☐	COBBLE [8]	☐	☐	____
☐	☐	GRAVEL [7]	☐	☐	____
☐	☐	SAND [6]	☐	☐	____
☐	☐	BEDROCK [5]	☐	☐	____

OTHER TYPES

PREDOMINANT P	R		PRESENT P	R	TOTAL %
☐	☐	HARDPAN [4]	☐	☐	____
☐	☐	DETRITUS [3]	☐	☐	____
☐	☐	MUCK [2]	☐	☐	____
☐	☐	SILT [2]	☐	☐	____
☐	☐	ARTIFICIAL [0]	☐	☐	____

(Score natural substrates; ignore sludge from point-sources)

NUMBER OF BEST TYPES: ☐ 4 or more [2] ☐ 3 or less [0]

Check ONE (Or 2 & average)

ORIGIN

☐ LIMESTONE [1]
☐ TILLS [1]
☐ WETLANDS [0]
☐ HARDPAN [0]
☐ SANDSTONE [0]
☐ RIP/RAP [0]
☐ LACUSTRINE [0]
☐ SHALE [-1]
☐ COAL FINES [-2]

QUALITY

SILT
☐ HEAVY [-2]
☐ MODERATE [-1]
☐ NORMAL [0]
☐ FREE [1]

SEDIMENT DEPOSITS
☐ EXTENSIVE [-2]
☐ MODERATE [-1]
☐ NORMAL [0]
☐ NONE [1]

Substrate Maximum 20

Comments

2] *INSTREAM COVER* Indicate presence 0 to 3 and estimate percent: **0**–Absent; **1**–Very small amounts or if more common of marginal quality; **2**-Moderate amounts, but not of highest quality or in small amounts of highest quality; **3**-Highest quality in moderate or greater amounts (e.g., very large boulders in deep or fast water, large diameter log that is stable, well developed root wad in deep/fast water, or deep, well-defined, functional pools.)

%	Amount		%	Amount		%	Amount	
____	____	UNDERCUT BANKS [1]	____	____	POOLS > 70cm [2]	____	____	OXBOWS, BACKWATERS [1]
____	____	OVERHANGING VEGETATION [1]	____	____	ROOTWADS [1]	____	____	AQUATIC MACROPHYTES [1]
____	____	SHALLOWS (IN SLOW WATER) [1]	____	____	BOULDERS [1]	____	____	LOGS OR WOODY DEBRIS [1]
____	____	ROOTMATS [1]						

AMOUNT
Check ONE (Or 2 & average)
☐ EXTENSIVE > 75% [11]
☐ MODERATE 25 - 75% [7]
☐ SPARSE 5 - < 25% [3]
☐ NEARLY ABSENT < 5% [1]

Cover Maximum 20

Comments

3] *CHANNEL MORPHOLOGY* Check ONE in each category (Or 2 & average)

SINUOSITY	DEVELOPMENT	CHANNELIZATION	STABILITY
☐ HIGH [4]	☐ EXCELLENT [7]	☐ NONE [6]	☐ HIGH [3]
☐ MODERATE [3]	☐ GOOD [5]	☐ RECOVERED [4]	☐ MODERATE [2]
☐ LOW [2]	☐ FAIR [3]	☐ RECOVERING [3]	☐ LOW [1]
☐ NONE [1]	☐ POOR [1]	☐ RECENT OR NO RECOVERY [1]	

Channel Maximum 20

Comments

4] *BANK EROSION AND RIPARIAN ZONE* Check ONE in each category for EACH BANK (Or 2 per bank & average)

River right looking downstream

L	R	EROSION
☐	☐	NONE/LITTLE [3]
☐	☐	MODERATE [2]
☐	☐	HEAVY/SEVERE [1]

L	R	RIPARIAN WIDTH
☐	☐	WIDE > 50m [4]
☐	☐	MODERATE 10-50m [3]
☐	☐	NARROW 5-10m [2]
☐	☐	VERY NARROW [1]
☐	☐	NONE [0]

L	R	FLOOD PLAIN QUALITY
☐	☐	FOREST, SWAMP [3]
☐	☐	SHRUB OR OLD FIELD [2]
☐	☐	RESIDENTIAL, PARK, NEW FIELD [1]
☐	☐	FENCED PASTURE [1]
☐	☐	OPEN PASTURE, ROWCROP [0]

L	R	
☐	☐	CONSERVATION TILLAGE [1]
☐	☐	URBAN OR INDUSTRIAL [0]
☐	☐	MINING/CONSTRUCTION [0]

Indicate predominant land use(s) past 100m riparian.

Riparian Maximum 10

Comments

5] *POOL/GLIDE AND RIFFLE/RUN QUALITY*

MAXIMUM DEPTH
Check ONE (ONLY!)
☐ > 1m [6]
☐ 0.7 - < 1m [4]
☐ 0.4 - < 0.7m [2]
☐ 0.2 - < 0.4m [1]
☐ < 0.2m [0]

CHANNEL WIDTH
Check ONE (Or 2 & average)
☐ POOL WIDTH > RIFFLE WIDTH [2]
☐ POOL WIDTH = RIFFLE WIDTH [1]
☐ POOL WIDTH < RIFFLE WIDTH [0]

CURRENT VELOCITY
Check ALL that apply
☐ TORRENTIAL [-1]
☐ VERY FAST [1]
☐ FAST [1]
☐ MODERATE [1]
☐ SLOW [1]
☐ INTERSTITIAL [-1]
☐ INTERMITTENT [-2]
☐ EDDIES [1]

Indicate for reach – pools and riffles.

Recreation Potential
(Circle one and comment on back)
☐ Primary Contact
☐ Secondary Contact

Pool/ Current Maximum 12

Comments

Indicate for functional riffles; Best areas must be large enough to support a population of riffle-obligate species:

☐ NO RIFFLE [metric = 0]

Check ONE (Or 2 & average)

RIFFLE DEPTH
☐ BEST AREAS > 10cm [2]
☐ BEST AREAS 5 - 10cm [1]
☐ BEST AREAS < 5cm [metric = 0]

RUN DEPTH
☐ MAXIMUM > 50cm [2]
☐ MAXIMUM < 50cm [1]

RIFFLE/RUN SUBSTRATE
☐ STABLE (e.g., Cobble, Boulder) [2]
☐ MOD. STABLE (e.g., Large Gravel) [1]
☐ UNSTABLE (e.g., Fine Gravel, Sand) [0]

RIFFLE/RUN EMBEDDEDNESS
☐ NONE [2]
☐ LOW [1]
☐ MODERATE [0]
☐ EXTENSIVE [-1]

Riffle/ Run Maximum 8

Comments

6] *GRADIENT* (ft/mi)
☐ VERY LOW - LOW [2-4]
☐ MODERATE [6 - 10]
☐ HIGH - VERY HIGH [10 - 6]

DRAINAGE AREA (mi^2)

%POOL: ____ %GLIDE: ____
%RUN: ____ %RIFFLE: ____

Gradient Maximum 10

IDEM 07/06/10

IDEM

OWQ Biological Studies QHEI (Qualitative Habitat Evaluation Index)

COMMENT _____

A-CANOPY
- ☐ > 85% - Open
- ☐ 55% - < 85%
- ☐ 30% - < 55%
- ☐ 10% - < 30%
- ☐ < 10% - Closed

B-AESTHETICS
- ☐ Nuisance algae
- ☐ Invasive macrophytes
- ☐ Excess turbidity
- ☐ Discoloration
- ☐ Foam/Scum
- ☐ Oil sheen
- ☐ Trash/Litter
- ☐ Nuisance odor
- ☐ Sludge deposits
- ☐ CSOs/SSOs/Outfalls

C-RECREATION

	Area	Depth
Pool:	☐ > 100 ft^2	☐ > 3 ft

D-MAINTENANCE
- ☐ Public ☐ Private
- ☐ Active ☐ Historic
- Succession: ☐ Young ☐ Old
- ☐ Spray ☐ Islands ☐ Scoured
- Snag: ☐ Removed ☐ Modified
- Leveed: ☐ One sided ☐ Both banks
- ☐ Relocated ☐ Cutoffs
- Bedload: ☐ Moving ☐ Stable
- ☐ Armoured ☐ Slumps
- ☐ Impounded ☐ Desiccated
- ☐ Flood control ☐ Drainage

E-ISSUES
- ☐ WWTP ☐ CSO ☐ NPDES
- ☐ Industry ☐ Urban
- ☐ Hardened ☐ Dirt & Grime
- ☐ Contaminated ☐ Landfill
- BMPs: ☐ Construction ☐ Sediment
- ☐ Logging ☐ Irrigation ☐ Cooling
- Erosion: ☐ Bank ☐ Surface
- ☐ False bank ☐ Manure ☐ Lagoon
- ☐ Wash H_2O ☐ Tile ☐ H_2O Table
- Mine: ☐ Acid ☐ Quarry
- Flow: ☐ Natural ☐ Stagnant
- ☐ Wetland ☐ Park ☐ Golf
- ☐ Lawn ☐ Home
- ☐ Atmospheric deposition

Looking upstream (> 10m, 3 readings; < 10m, 1 reading in middle); Round to the nearest whole percent

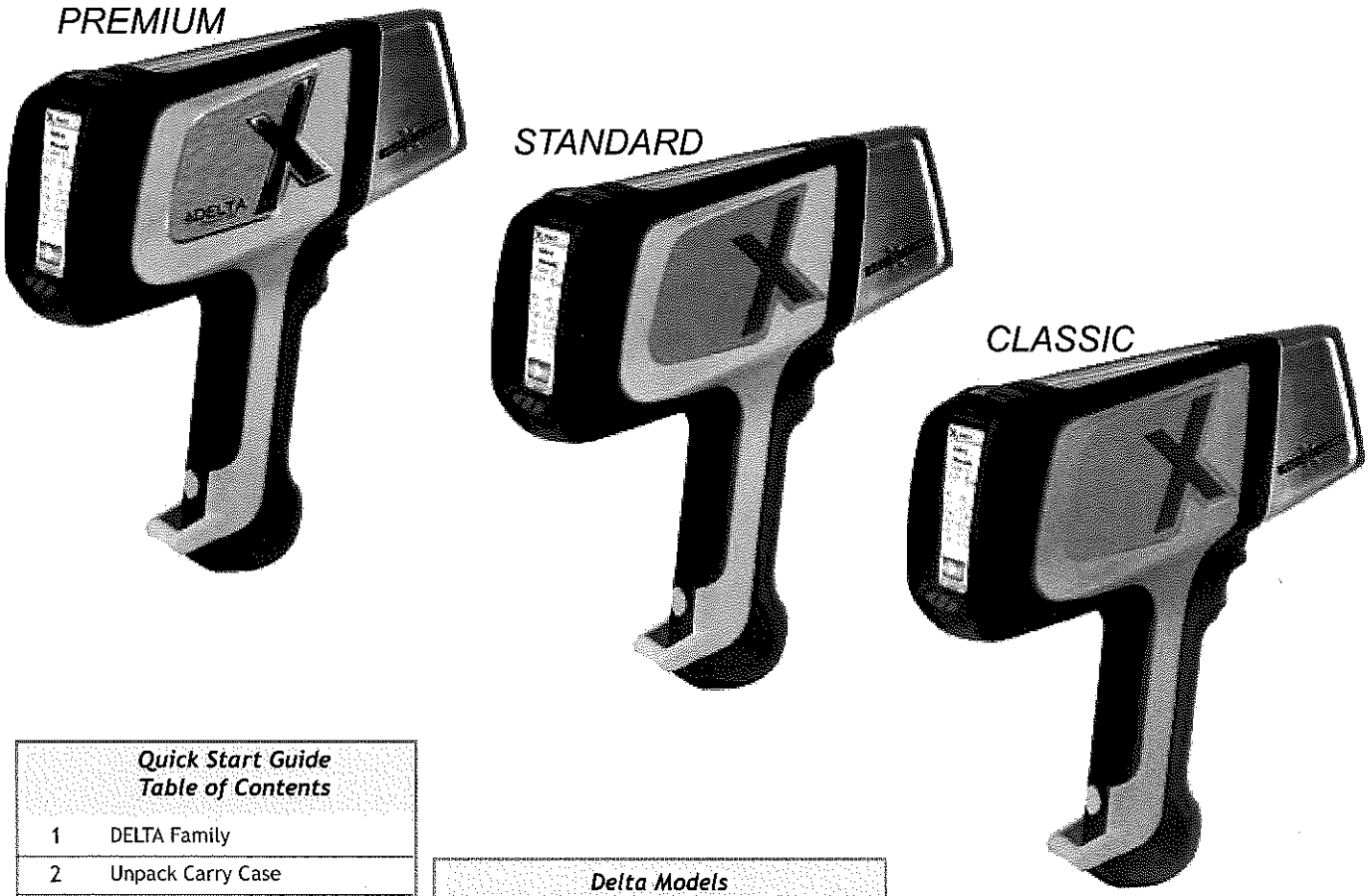
	Left	Middle	Right	Total Average
% open	____ %	____ %	____ %	____ %

Stream Drawing:

Attachment B – Manufacturer's Manuals

Quick Start Guide

DELTA Family of Handheld XRF Analyzers



Quick Start Guide Table of Contents

1	DELTA Family
2	Unpack Carry Case
3	Tour: Analyzer
4	Tour: Docking Station
5	Components & Accessories
5	Return Procedures
6	Safety Information
7	Warning Label & Indicators
8	Prepare for Operations
8	Battery Issues
9	Snapshot of User Interface
10	GUI Elements: Icons/Buttons
11	Typical Operating Procedures
12	Append A: Radiation Profile

Delta Models

Type	Modes	Models
Premium	Alloy	DP-2000
	Environmental	DP-4000
	Mining	DP-6000
	RoHS	DP-6500
Standard	Alloy	DS-2000
	Environmental	DS-4000
	Mining	DS-6000
	RoHS	DS-6500
Classic	Alloy	DC-2000
	Environmental	DC-4000
	Mining	DC-6000
	RoHS	DC-6500

DELTA
Dynamic XRF

PN 103076
Rev.3.0: 2/2011

Unpack the Instrument and Docking Station

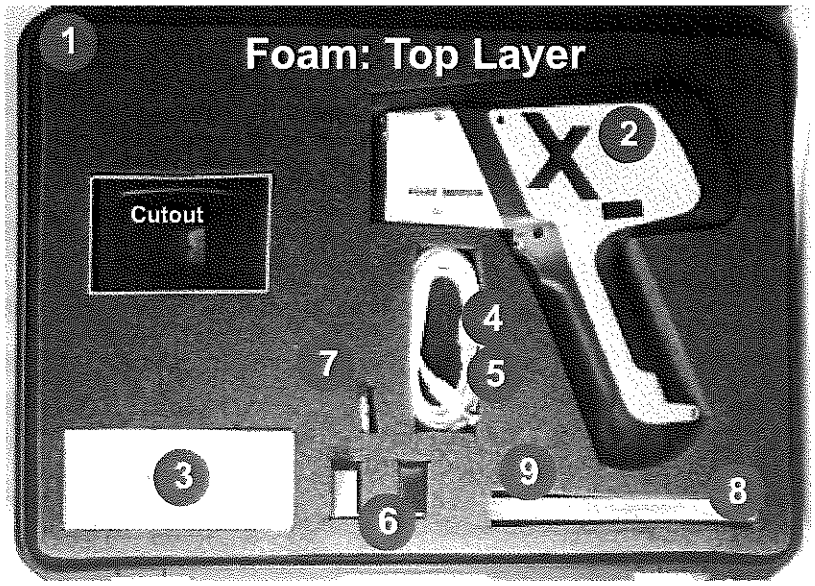
To unpack the instrument:

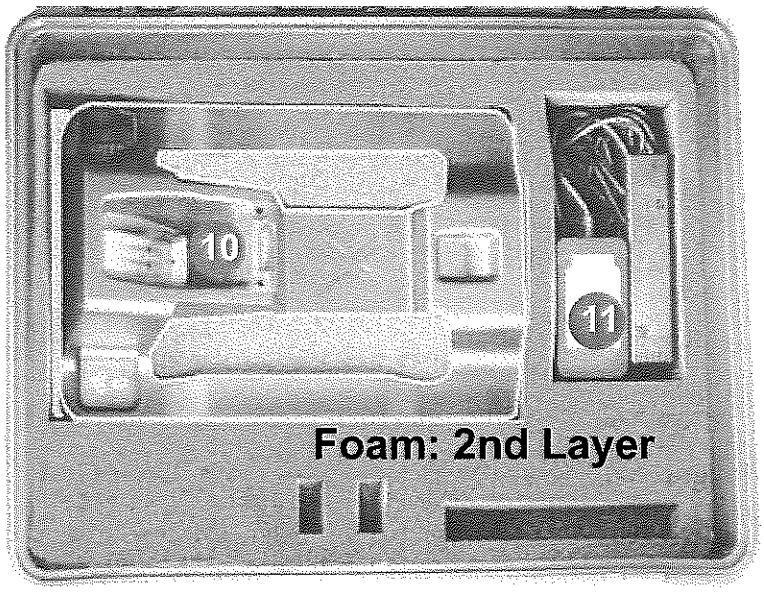
1. Locate and remove the shipping papers and documentation.
2. Open the Carrying Case and remove the Delta and all of the components
3. Note that the protective foam has **TWO LAYERS.**
4. With the Foam Top Layer clear of items, lift it to expose the Docking Station and the optional AC Power Adapter
5. Inspect all components for damage and report any to Innov-X immediately.

ITEM CHECKLIST for DELTA Handheld XRF Analyzer

Component Key

	—Foam: Top Layer—
1	Carry Case
2	Delta Analyzer
3	Docking Station Charger
4	USB Cable #1 (See pg 5 for details)
5	USB Cable #2 (See pg 5 for details)
6	Li-ion Batteries (2)
7	Cal Check Coupon
8	Extra Windows (Bag of 10)
9	End/User Documentation
	—Foam: 2nd Layer—
10	Docking Station
11	AC Power Adapter (Optional)



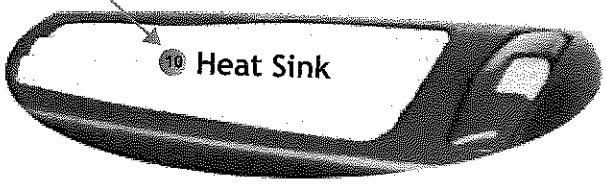


Tour of Instrument

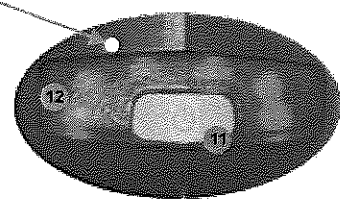
1. Handheld Analyzer

Component Key

	Delta – All Models
1	Delta Analyzer (Premium Model Shown)
2	Probe
3	Measurement Window (Prolene Film)
4	Hinged Window Plate
5	Docking Station Connector
6	Trigger
7	Handle – Non-Slip Rubber Grip
8	Battery Boot
9	Data Port w/ Rubber Cover
10	Heat Sink
11	I/O (Power) Switch w/ LED Indicator
12	X-ray Warning Light Array
13	Touchscreen for User Interface
14	Navigation Keys



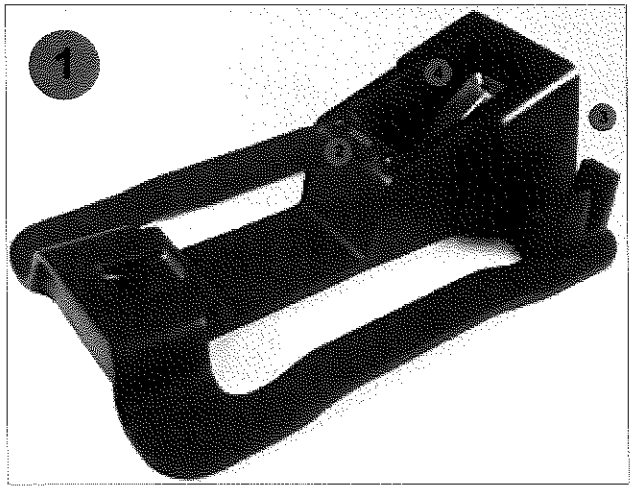
10 Heat Sink



11 I/O Power Switch w/LED Indicator

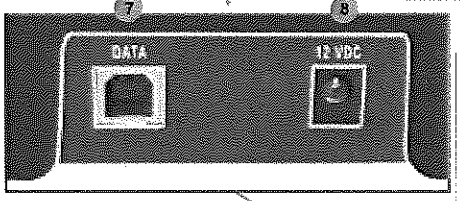
12 X-ray Warning Light Array

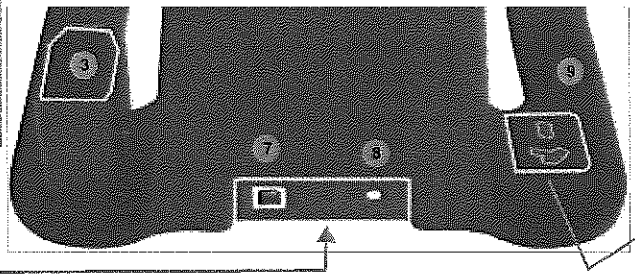
2. Docking Station

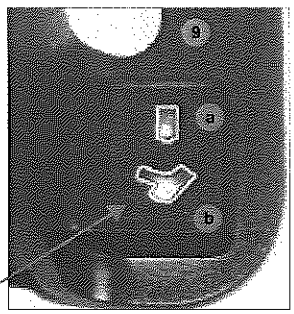


Component Key

	Delta – All Model
1	Docking Station (Empty)
2	Analyzer Signal/Control Connector
3	Second Battery Charge Socket
4	CalCheck Test Cup (316 stainless steel)
5	Docking Station (Loaded)
6	Second Battery in Socket
7	Data Port(s): — Docking Station –>Rear
	— Analyzer –> Left Side
8	Input Power (12 VDC)
9	Indicator Lights
a	Second Battery Charging
b	Analyzer Engaged



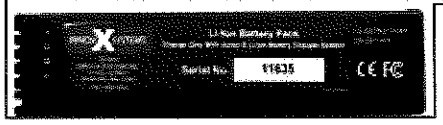




3. Components and Accessories

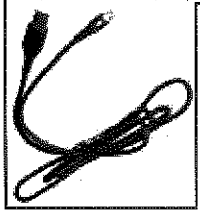
Items included with a Delta analyzer and docking station are shown below.
Unless otherwise noted, all parts are standard accessories.

Li-ion batteries
High capacity, 4.8-5.2 Ah; two provided (one shown).



USB Cable #1
Six feet, 480 Mbps, USB A to USB B connectors.

USB Cable #2
Mini B to USB A connector.

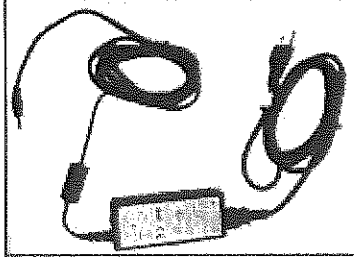


Kapton Windows - Classic Model only
Bag with 10 pieces of 6 µm film windows.
(Not shown)

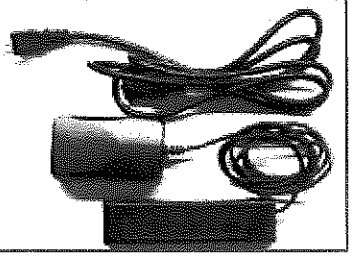
CalCheck (Standardization) Coupon.

Prolene Windows - Premium & Standard Models
Bag with 10 pieces of 6 µm film windows.
(Not shown)

AC Power Adapter
Provides DC power to Docking Station;
Input: 110-240 VAC; Output: 60 W, 12 VDC, 5 A.



AC Power Adapter
Li-ion battery replacement; 110 - 240 VAC power source. This unit is an **Optional Accessory**.



Delta Documentation Suite
User Manual (P/N 103201)
User SW Interface Guide (P/N 103202)
(Not shown)

Safety and Informational Package
(Not shown)

Packing and Shipping

If the instrument is not returned in the protective case, it can be damaged during shipping. Innov-X Systems reserves the right to void the warranty on instruments that are damaged during return shipping that are sent without the protective case.

Prior to returning a unit, customers must contact Customer Service at 1-781-938-5005 or technicalsupport@innovxsys.com to receive the required RMA number and to answer any shipping questions.

Follow these instructions to return your XRF Analyzer:

1. Pack the analyzer in the black protective case in which it arrived, using the original packing materials.
2. Include the RMA in the case and reference the RMA number in your shipping documents.
3. Close the protective case and either:
   - Secure it with plastic zip ties,
     — or —
   - Pack the protective case within another box.

Radiation Safety Information

Innov-X's Handheld XRF Analyzers are secure and dependable instruments when used according to recommended testing techniques and safety procedures.

WARNING

- **Innov-X analyzers must be used by trained and authorized operators, according to proper safety procedures. Improper usage may circumvent safety protections and could potentially cause harm to the user.**
- **Heed all warning labels and messages.**
- **DO NOT USE the instrument if there is any chance that it is damaged or might unintentionally emit stray radiation. In such a case, arrange for qualified personnel to perform a radiation safety test and repair any analyzer damage.**

Safety Interlock Structure

For controlling an Innov-X handheld instrument's X-ray emissions, and therefore minimizing the possibility of accidental exposure, there is a standard safety interlock structure consisting of the three features listed below.

1. Software Proximity Sensor

- Within one second of a test start, the analyzer will detect a sample in front of the measurement window. If not, to prevent accidental exposure, the test aborts, the filter wheel goes to position 0, and the x-rays shut off. The tube current is reduced to 0.0 microAmperes and the red light stops blinking. Also when a test is in progress, if the probe/nose is pulled away from the sample, the test stops in approximately one second.

2. Software Trigger Lock

- If five minutes elapse between tests (default time), the trigger locks automatically and you must tap on the lock icon to unlock it.

3. Safeguards

As an owner of an Innov-X handheld XRF instrument your safeguards include those items noted below:

Limited Access

Keep the instrument in a controlled location, where only trained and authorized users are likely to have access.

Trained Operators

Keep a sign with the analyzer indicating that in order to use it an operator must have completed a training class provided by your company, or must have attended an Innov-X training course and completed any other requirements as dictated by the local regulating authority. When the Innov-X system is turned on, the controller screen displays a message indicating that the system should only be used by authorized personnel.

Shielding Issues

BACKGROUND:
An Innov-X handheld XRF instrument emits a tightly collimated beam of X-ray radiation. Although attenuation occurs, the beam may project many meters in open air.
ACTION:
Adequate shielding is achieved by:

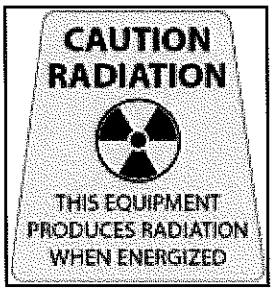
- Establishing a *no-admittance zone* sufficiently distant from the instrument's measurement window that allows air to attenuate the beam.
- Enclosing the beam working area with protective panels (for example, 3.0 mm stainless steel can attenuate the beam to background levels)

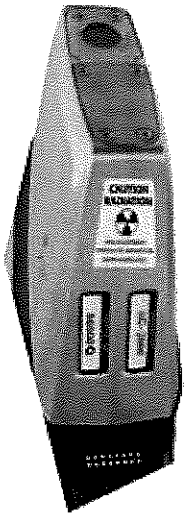
Contact your Innov-X Systems representative for assistance and suggestions on interlocks and applications for limiting radiation exposure.

Trigger Issues

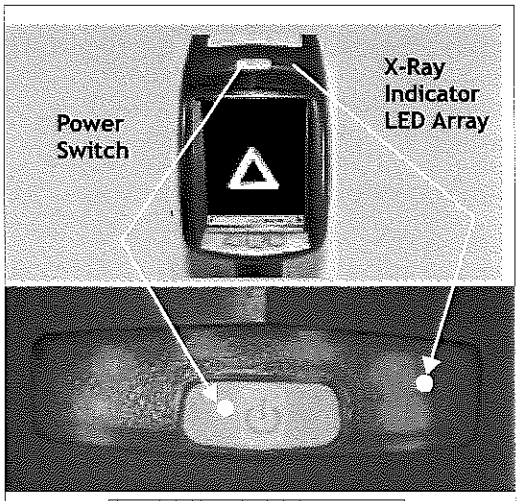
"Deadman trigger" mode requires the user to PULL AND HOLD the trigger for the DURATION of the test. Releasing the trigger immediately aborts the test.

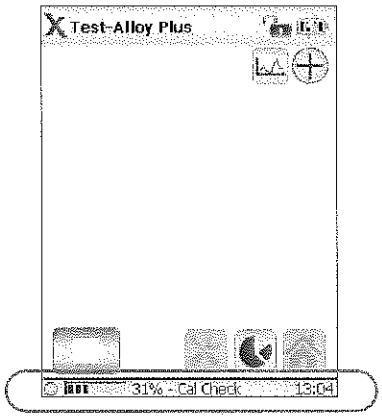
Warning Label and Indicators





Caution Radiation Label:
Underneath Probe/Nose





Indicators and Status

Power Switch with Integral Indicator Light

The power switch is located at the upper rear of the unit.

POWER ON

- Press the I/O switch to turn on the power.
  - A green LED indicator comes on.
- This switch ***DOES NOT*** turn on the x-ray tube.
  - No tube power is supplied until the Innov-X software is launched.

SLEEP

- With the unit running, press I/O switch for an instant.
  - Analyzer goes into a "Sleep" state; screen is off; no further activities are possible.
  - The Green LED shows a **variable intensity**, going from dim to bright to dim in a repeating fashion.
- Press the switch for an instant again to restore the UI screen.

POWER OFF

- Press and hold switch for >3 seconds.
  - Unit powers off. (See page 11 for more Exit options)

UI Screen Note

All User Interface screens have a **time-out** (power-saving) feature that causes a screen to go blank if the UI is not accessed or the unit is not moved after a **90** second interval. However, the analyzer is still running.
Restore the screen by tapping it or by moving the unit.

X-Ray Indicator

The X-ray indicator is located on the upper rear of the unit.
It consists of a six red LED array.
It provides TWO KEY FUNCTIONS:

1. X-RAY INDICATOR ON CONTINUOUSLY (*SOLID RED LED ARRAY*)
   This signifies:
   - X-ray tube is enabled.
   - There is no radiation exposure to you or bystanders.

   The instrument can be carried or set down safely in this condition.
2. X-RAY INDICATOR ON FLASHING (*BLINKING RED LED ARRAY*)
   This signifies:
   - X-ray tube is powered to full operational level.
   - Analyzer is emitting x-ray radiation through the analysis window.

   In this condition, the analyzer must be pointed at a test sample.

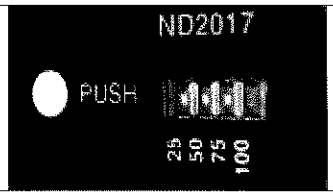
Test Screen

When making a CalCheck or testing a sample, the TEST screen's lower status bar provides an **indication of the progress.**
Upon completion, message indicates **Ready** (for next operation)

PREPARATION for Operations - Battery Issues

1. Battery Status

To test a Li-ion Battery's charge status, press the white button on the battery. The green lamps indicate the percent of charge, from less than 25% to 100%. If a battery has a charge of less than 25%, use the Docking Station to establish a full charge.

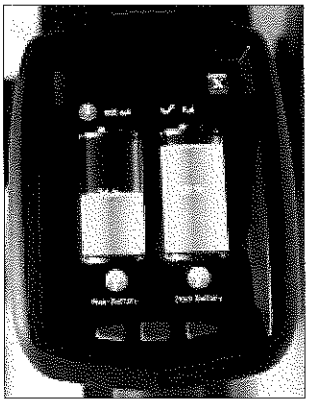


2. Charging Batteries

The Delta analyzer has a new multi-purpose tool: a **Docking Station.** {See Page 4 for hardware details}
In addition to providing an automatic Calibration Check, the Docking Station delivers **two** charging functions:

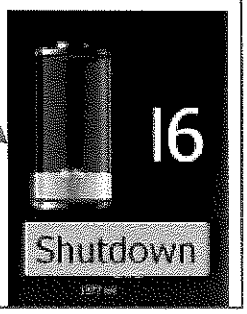
- It can charge the installed Li-ion battery located in the instrument's **handle.**
- Simultaneously, it can charge a second battery with its special battery charging **socket.**

Charge status is shown in real-time on the Delta's display screen.
The second docked battery's status (either "charging = red" or "full = green") is also shown on the battery icon located on the rear left side of the Docking Station.



3. HOT SWAP for a Delta Battery

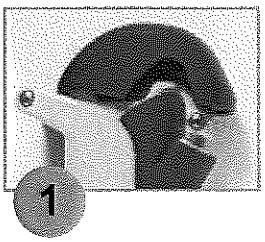
A battery Hot Swap capability is a standard feature with the Delta analyzer. An operator can remove and replace a battery without having to shut down, restart, or Cal Check.
A "Shutdown" status display gives the percentage of internal charge remaining when the battery is removed.
If the internal charge reaches 0 you have to re-start the unit with the I/O switch, after inserting a fresh battery.
If red X-ray indicator lights flash, the battery voltage is too low.

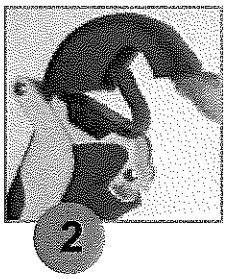


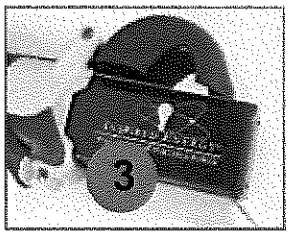
4. Changing a Battery

To CHANGE the battery:

1. Hold the instrument by the handle, upside down, so the bottom of the instrument base is pointing upward with the nose pointing away from the operator.
2. Pull the rubber latch and lift cover.
3. Remove the existing battery using the tab.
4. Insert the charged battery into the analyzer with the battery connectors facing to the left. The battery slot is keyed so that the battery can be inserted only one way.

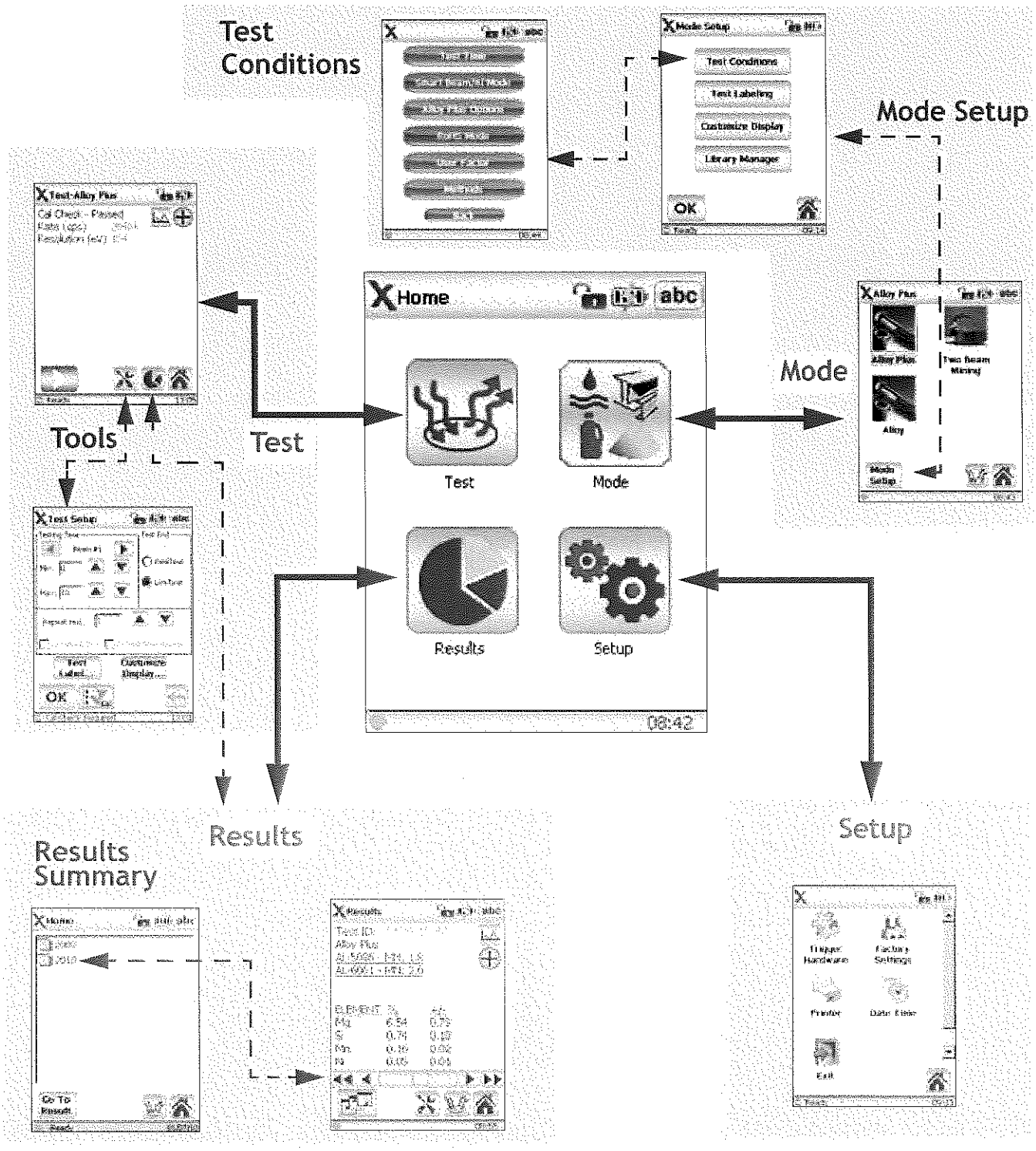






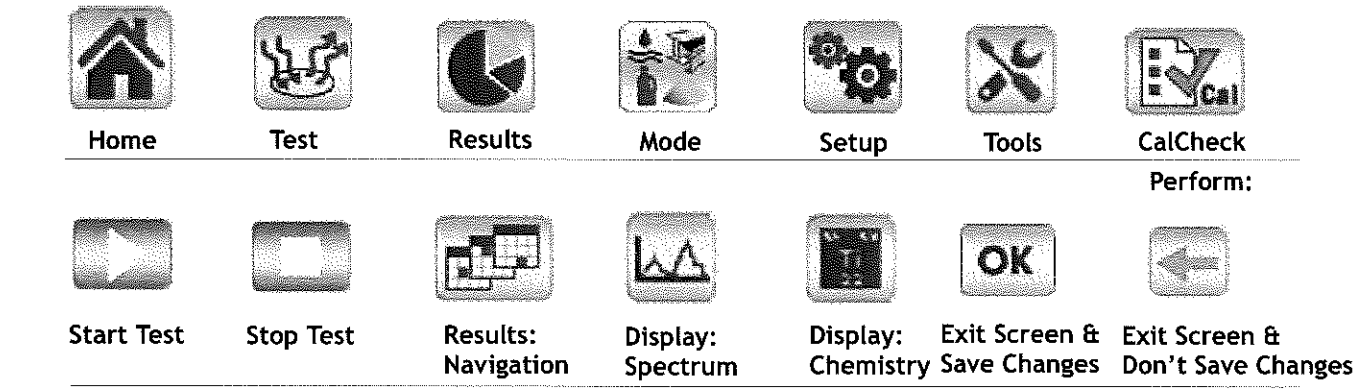
SNAPSHOT: Delta User Interface

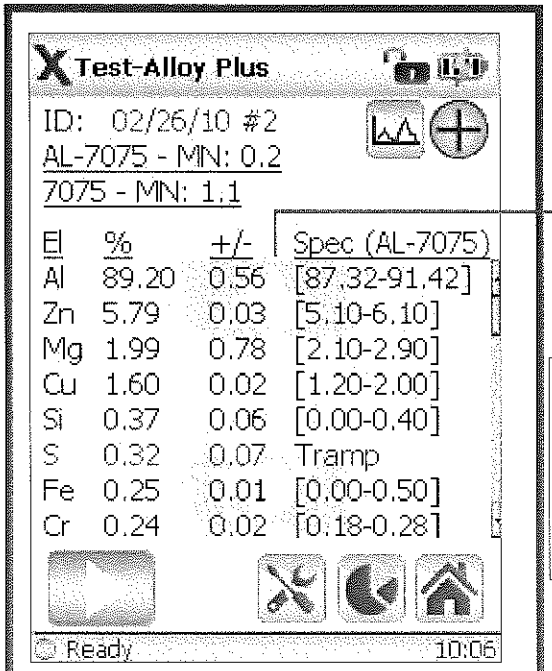
The Delta's user interface is introduced by the startup **Radiation Safety** and **Initialization** screens. {*See page 11*}
Main operations then revolve around the **Home** screen.



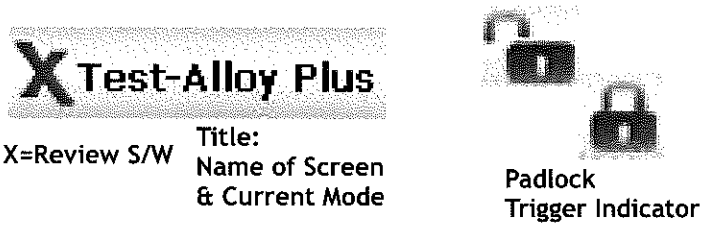
GRAPHIC ELEMENTS: Delta User Interface

ICONS and BUTTONS and INDICATORS





Typical User Interface Screen



X=Review S/W

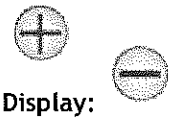
Title: Name of Screen & Current Mode

Padlock Trigger Indicator

Display: Keyboard

Battery Status

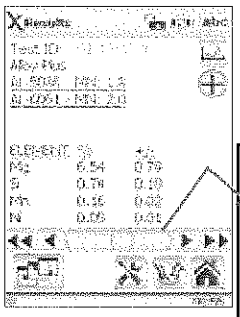
On Screen *Vertical* Scrolling



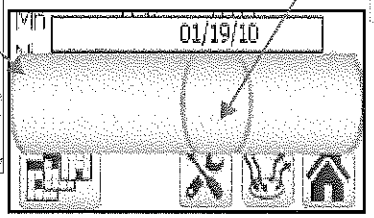
Display: Expand

Display: Minimize

Information Indicator:
Black - Messages
Red - Errors

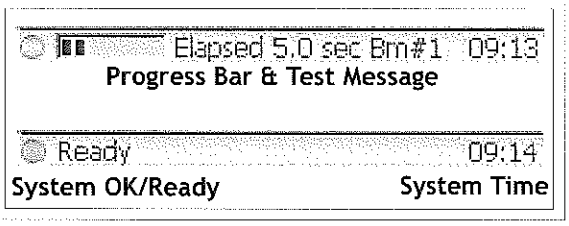


On Screen *Horizontal* Scrolling



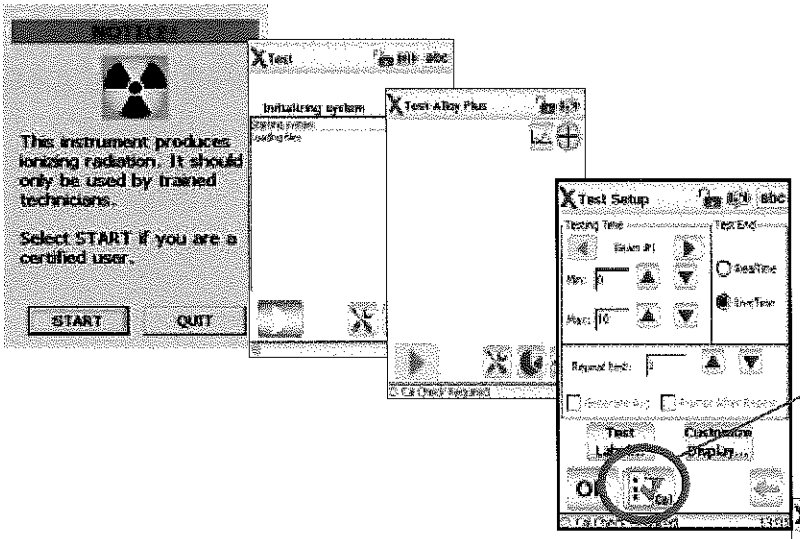
Use finger to move display vertically — or — horizontally for added information

Lower Status Bar



Go to *User S/W Interface Guide*, P/N 103202, for complete S/W details.
(Available: February, 2011)

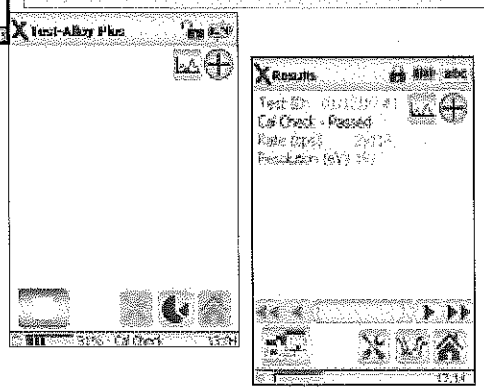
Typical Operating Procedure



About CalCheck:

Delta analyzers use advanced techniques for calibration checking.

- The Docking Station (with its special 316 s/s probe cup) provides an automatic Cal Check. The instrument must be ON when inserted.
- In the field (away from your Docking Station) you can use the 316 s/s standardization coupon to do a CalCheck. The check only takes 15 seconds.



A typical operational sequence:

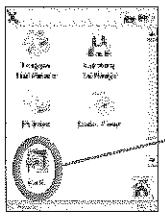
1. Insert a charged battery in analyzer handle.
2. Turn on instrument with I/O switch
3. Read the radiation safety notice screen and acknowledge that you are a certified user.
4. System Initialization begins immediately
5. Unit will launch a Test screen using the Mode last selected.
   - **5a.** If the mode should be changed, go to Home screen.
   - **5b.** Select the Mode button
   - **5c.** Choose the desired Mode
6. When message *Cal Check Required* is present, place unit in the Docking Station; navigate to Test Setup, tap Cal Check button, tap Start Test button. CalCheck takes about 15 seconds.
7. After a successful Cal Check, unit is ready to use.
8. Position it's measurement window over the test sample.
9. Pull the trigger or tap the Start Test button.

Results are displayed immediately at test completion.

10. Choose the Spectrum icon to view special results.
11. At end of a testing session, export results to a PC using data port and USB cable.
12. When testing and exporting are complete, turn off Delta with I/O switch or place in Docking Station.

Go to page 7 to review Standby and Time-Out Features

Exit Options



Setup Screen

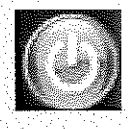
Exit

Confirm

User has **three** options:

Soft Reboot
Restart OS and restart IX app.

Power OFF
Turn analyzer off after confirmation.

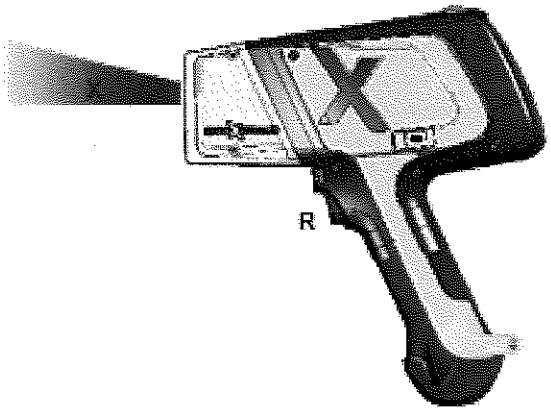


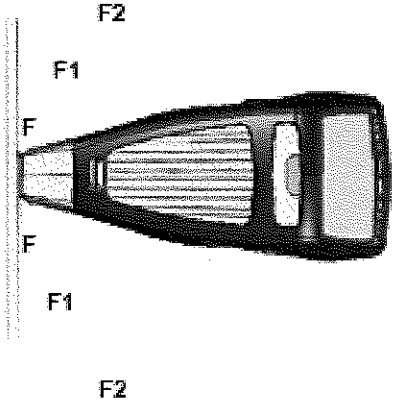
Relaunch
Restart IX app.

Appendix A. DELTA Radiation Profile

This is the current Delta Radiation Profile:

RADIATION PROFILE – Hand Held Instruments





Measured Shallow Dose Rate in mR/h - Secondary Radiation (Scatter)

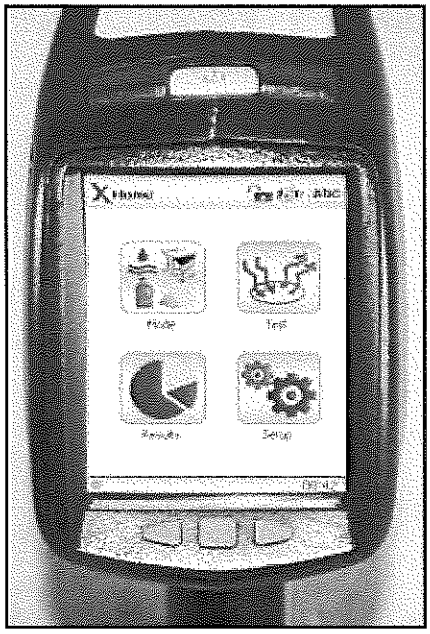
Mode(s)	Substrate	Voltage kV	Amperage µA	Filter	Trigger R	Close F	10 cm F1	30 cm F2
Alloy Plus Mining 2-Beam Mining	316 Stainless	40	100	1	0.1	1	0.5	0.1
	Al (319 AA)				0.2	6	3	0.6
	Soil (SiO_2)				3	50	25	9
Alloy Plus 2	316 Stainless	13	200	7	BK	0.1	BK	BK
	Al (319 AA)				BK	BK	BK	BK
2-Beam Mining 2	Soil (SiO_2)	10	200	7	BK	0.1	BK	BK
3-Beam Soil 1		40	100	3	1	16	7	2
3-Beam Soil 2		40	100	2	3	100	25	4.5
3-Beam Soil 3		15	200	5	BK	0.1	BK	BK

BK – No higher than background

TEST CONDITION: Instrument run at normal setting for mode and represents typical production unit.

User Manual

Delta™ Family: Handheld XRF Analyzers



PN_103201
Rev_A: June/2010

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Revision History

Release Date for this document and its individual sections is June, 2010. This enters the Innov-X document control system as Revision A

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GO TO

See "*A7. Legal Information*" for information concerning Innov-X Systems, Inc.'s warranties, licenses, and liabilities.

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Delta Family End User Documentation Resources

During Delta's development and initial product shipments several End User documents have been created. They are listed in the table below.

Delta Documentation Resources		
Innovx Part #	Release Date	Title
103202_RevA	July/2010	Delta User Interface Guide (UI version 2.5))
103201_RevR	June/2010	Delta Family User Manual (This document)
103076_RevA	3/2010	Delta Family Quick Start
101593_RevA	11/2007	Window Replacement: Hinged Plate HandHeld Analyzers
102922_RevA	2/2010	Delta Family User Manual (Canadian Edition)
103158_RevA	3/2010	HOW TO: Setup and Configure A-020-D Teststand/Workstation for Delta Analyzer
TBA	6/2010	HOW TO: Convert A-020-A or A-020-O Teststand/Workstation to Support a Delta Analyzer

Preface

This *Preface* provides the following information:

- "Manual Structure"
- "Document Conventions"

Manual Structure

This *User Manual* consists of eight chapters, ten appendices, this Preface, Table of Contents, and a Cover. Individual chapter material is summarized below:

C1. Introduction describes the basics of the system:

- The Innov-X Delta™ Family
- Visual tour of the instrument noting all the major components

C2. Safety Information describes general safety information:

- Priority Information
- General Precautions
- Electrical Precautions
- X-ray Safety
  - Safety Interlock Structure
  - Safe and Unsafe Usage Scenarios
  - Radiation Doses for Several Scenarios
  - Comparative Analysis of Typical Exposure
  - Common Questions and Answers
- Delta Radiation Profile
- Required Certification
- Analyzer Shut-down Procedures

C3. Safety Administration describes safety program information:

- Radiation Safety Training Recommendations
- Dosimeter badges
- A typical dosimeter monitoring program
- Dosimeter service contractors
- Registration requirements

C4. Operations describes operations and testing procedures:

- Configure and Use Delta Docking Station (DDS)
- Start-up Procedure
- Cal Check Issues
- Battery Issues
- Conducting and Ending Test Operations

NOTE

Information concerning the Delta Family's user interface is supplied in the companion document *"Delta User Interface Guide"* (PN103202_Rev2.5 June/2010).
The goal is to provide revised Delta UI Guides when a substantial software change is released.

C5. Alloy Analysis Modes describes five specific modes and calibrations including:

- Alloy
- Alloy Plus
- FastID
- Pass/Fail
- Precious Metal Additions

C6. Mining Analysis Modes describes three specific modes and calibrations including:

- Mining
- Two Beam Mining
- Car Catalyst

C7. Soil Analysis Modes describes two specific modes and two calibrations including:

– Soil	– Environmental
– Three Beam Soil	– Exploration

- LEAP issues for Classic Delta (PiN detector)
- Check Standards
- Sample Preparation

C8. Consumer Goods Modes describes two specific modes including:

– RoHS	– Consumer Products

- *RoHS Mode* provides a details from EU regulation directives which list the limits for RoHS elements and information for qualitative measurements.
- *Consumer Products Mode* is dedicated to testing for Lead (Pb)

A1. Overview: X-Ray Fluorescence (XRF) Spectrometry presents background information and general knowledge, including:

- Basic Theory and X-ray History
- Elemental Analysis
- EDXRF Spectrometers

A2. Soil Testing presents information on using the analyzer for soil analysis within certain accepted guidelines, including:

- Status for Field Portable XRF and Overview of Field Usage
- Quality Assurance
- Calibration for Innov-X Portable XRF
- Effects of Moisture on XRF Results
- Comparing XRF Results to Laboratory Results
- Common Interferences
- Sample Prep Procedures and Testing Protocols

A3. Specifications presents analyzer hardware and software specifications.

A4. Typical Delta Test Sequence

- Prerequisites noted by Mode
  - Grade Libraries
  - Check Standards
  - Sample Presentations
- Typical Test Sequence

A5. User Maintenance provides a key procedure/technique:

- Using the AC Power Adapter kit to replace a Li-ion battery
- Using the stand-alone battery charger
- Replacing a Prolene, Mylar, or Kapton Window

A6. Packing and Shipping gives the procedure for returning a unit to Innov-x.

- Warning Label for shipping products with Li-ion batteries

A7. Legal Information presents material, including:

- Analyzer Limited Warranty including:
- Limitation of Liability
- Warranty Period, Returns, and Repairs
- Instructions for Contacting Innov-X
- End User Software License Agreement including:
  - Use, Restrictions, and Termination of Software
  - Liability Limitations

A8. Alloy Grade Libraries including the Alloy Factory Grade library for each Model, and a "Tramp" Library with seven base alloys.

Document Conventions

Messages

There are four messages used in this Manual:

WARNING, CAUTION, NOTE, and **GOTO.**

They are characterized by an icon and a message box topped with a colored banner. The message text is on a gray background. An example of each message is below:

WARNING

DEMANDS that you observe the actions given in the text.
The **WARNING** message has a **bold** type style.

Remember:
The WARNING icon signifies information that denotes a potentially hazardous situation, which if not avoided, may result in serious injury or death.

CAUTION

SUGGESTS that you review the referenced details and heed the instructions offered.
The CAUTION message has a regular type style with *emphasized keywords.*

NOTE

REQUESTS that you pay particular attention to a specified procedure or piece of information. Adds details that make it easier to use the system and this manual.
The NOTE message has a regular type style.

GO TO

DIRECTS the user to another portion of this manual, or to other reference materials containing relevant data.
The **GOTO** (or Pointer) message has a regular type style.

Type Styles

These conventions are used to present information:

Convention (Type Style)	Description
Bold	Indicates an action taken on a button or other item.
Italic	Menu commands, names of keys, buttons, tabs, or items from picklists. User-entered text. It is used for references to other documents, C(hapter) titles, and A(ppendix) titles (for example, "... see "*C2. Safety Information*"). Labels on unit's I/O panels; panel or window names of the UI (User Interface).
`Courier` type-face	Computer displayed text or filename.

Pagination

Page numbering in this Manual is consecutive with the Front Cover being assigned Page Number 1. This enables the PDF document file and any hard-copy print to map to the Page field information in the Adobe Reader.

C1. Introduction

C1 includes a:

- Description of the Innov-X ***Delta***™ family of handheld XRF analyzers.
- Visual tour of the instrument(s) noting all the major features, and accessories.

Description of *Delta* System

What Is It?

The ***Delta*** is a handheld energy dispersive X-Ray fluorescence spectrometer, generally referred to as an XRF analyzer.A complete **Delta** package consists of:

- Handheld analyzer using an integrated group of instrument components that are sealed in an ergonomically designed, light-weight body. They include --
  - Controller
  - Color touchscreen (ergonomically mounted interactive display)
  - Membrane navigation keys
  - Choice of detectors (PiN or SDD) to meet wide-ranging application goals

Coordinated with these robust characteristics, the instrument's ***key feature*** is Innov-X's proprietary control, data acquisition, and analysis software with customer configured options.

Additional accessories (standard and optional) include:

- Li-Ion batteries (2) - {Standard}
- Delta Docking Station (DDS) Dedicated charging and calibration unit - {Standard}
- Rugged waterproof carry case - {Standard}
- Portable test stand to create a Delta workstation (A-020-D) {Optional}
- Soil foot (A-035) (Optional)
- Soil extension pole (990055) (Optional)
- Trimble Xplorer Package (Optional)

What Does It Do?

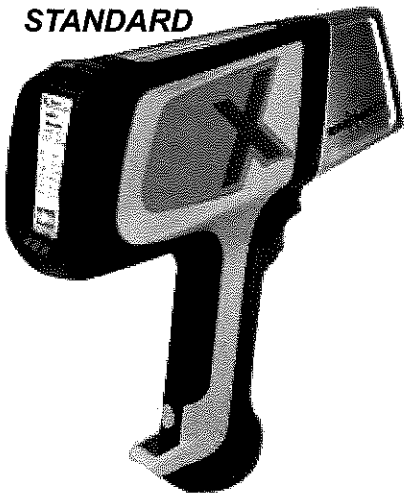
The expanded ***Delta*** family of handheld XRF instruments delivers fast and precise identification and analysis for elements from magnesium to uranium (Mg to U) depending on the selected model. A weatherproof/dustproof ultra rugged design including an integral heat sink permits users to conduct diverse analysis testing under severe operating conditions. An added convenience feature for field use is battery "Hot Swapping."

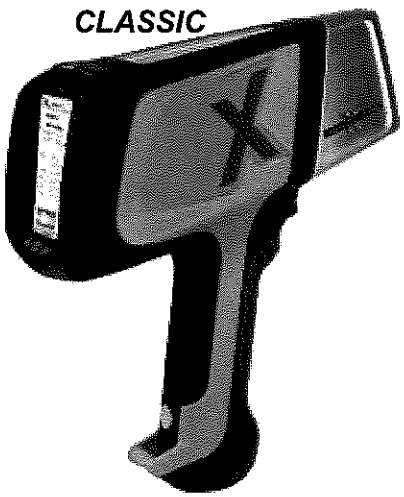
Applications

The analyzer gives accurate chemical analysis for commercial or industrial areas, such as:

- Positive Material Identification
- Mining and Exploration
- Consumer Safety
- Scrap Processing
- Environmental Testing
- Light Element & Aluminum Analysis

Delta Family: Types, Models, Modes and Calibrations





Delta Types and Models

Type	Modes	Models
Premium	Alloy	DP-2000
	Environmental	DP-4000
	Mining	DP-6000
	RoHS	DP-6500
Standard	Alloy	DS-2000
	Environmental	DS-4000
	Mining	DS-6000
	RoHS	DS-6500
Classic	Alloy	DC-2000
	Environmental	DC-4000
	Mining	DC-6000
	RoHS	DC-6500

Modes and Calibrations

ALLOY Analysis	Alloy
	Alloy Plus
	FastID & Pass/Fail
	Precious Metals
SOIL Analysis	Environmental
	Exploration
MINING	Mining Mode
	2 Beam Mining
	Car Catalyst
CONSUMER GOODS	RoHS
	Consumer Products
LEAD PAINT	Lead in Paint (HUD)
	Lead in Paint (Industrial)
THIN	Filter Analysis
	Dust Wipe

Inspection

Inspection

Use this procedure:

1. Remove the carry case from the shipping cartons; save cartons.
2. Open the carry case
   Remove the shipping documentation
3. Verify that all the parts and accessories are included.
   Remember that the case has **TWO FOAM LAYERS**.
4. Verify that no visible damage occurred during shipping.

WARNING

If there is damage to any of the components, DO NOT attempt to use the instrument.
Immediately contact Innov-X Customer Support at:

- **United States: 1-781-938-5005**
- **Europe: +31 (0)73 62 72 590**
- **Canada: 1-778-960-6279**
- **Australia: 02-9577-9500**

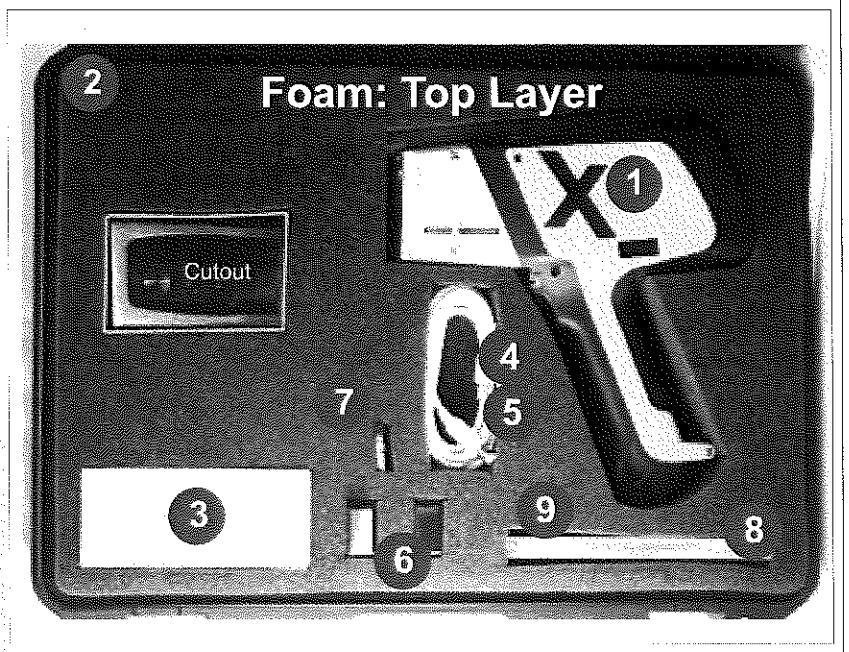
Or call your local distributor.

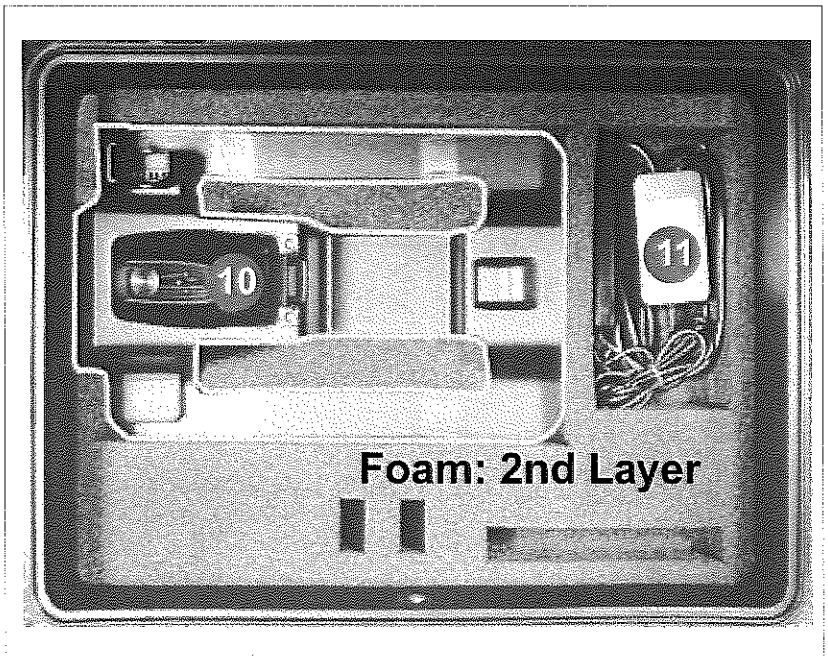
GO TO

- See *C4. Operations, Page 45 & 50 Battery Issues* for battery charging information.
- See *"Delta User Interface Guide"* (PN 103202) for a complete description of the *Innov-X* application's User Interface.
- See *A7. Legal Information* for warranty, liability, and software licensing information.

Tour of the *Delta* Package

The figure below depicts ***Delta's*** major components as initially shipped to a customer. Note that the protective foam in the Carry Case has **TWO LAYERS**.





Component Key

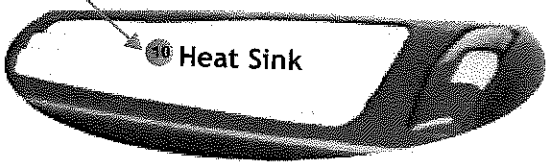
	—Foam: Top Layer—
1	Delta Analyzer
2	Carry Case
3	Docking Station Charger
4	USB Cable #1
5	USB Cable #2
6	Li-ion Batteries (2)
7	Cal Check Coupon
8	Extra Windows (Bag of 10)
9	End/User Documentation
	—Foam: 2nd Layer—
10	Docking Station
11	AC Power Adapter (Optional)

Tour of Instrument

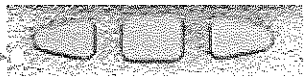
1. Handheld Analyzer

Component Key

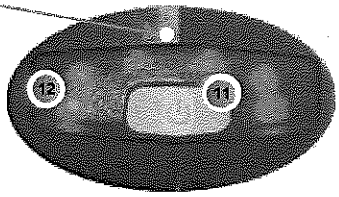
	Delta – All Models
1	Delta Analyzer (Premium Model Shown)
2	Probe
3	Measurement Window (Prolene Film)
4	Hinged Window Plate
5	Docking Station Connector
6	Trigger
7	Handle – Non-Slip Rubber Grip
8	Battery Boot
9	Data Port w/ Rubber Cover
10	Heat Sink
11	I/O (Power) Switch w/ LED Indicator
12	X-ray Warning Light Array
13	Touchscreen for User Interface
14	Navigation Buttons



10 Heat Sink



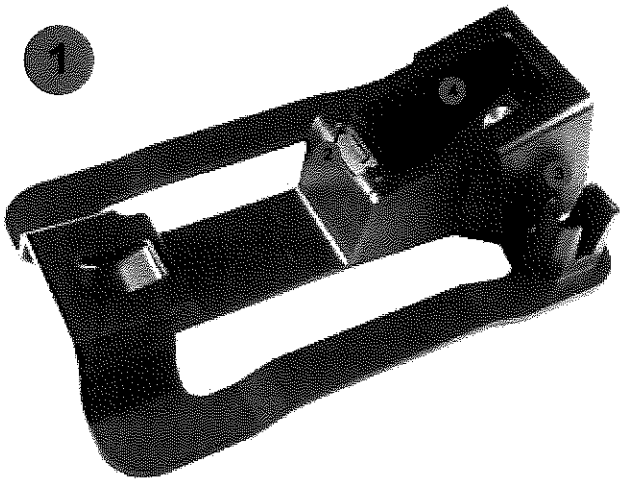
14 Navigation Buttons



11 I/O Power Switch w/LED Indicator

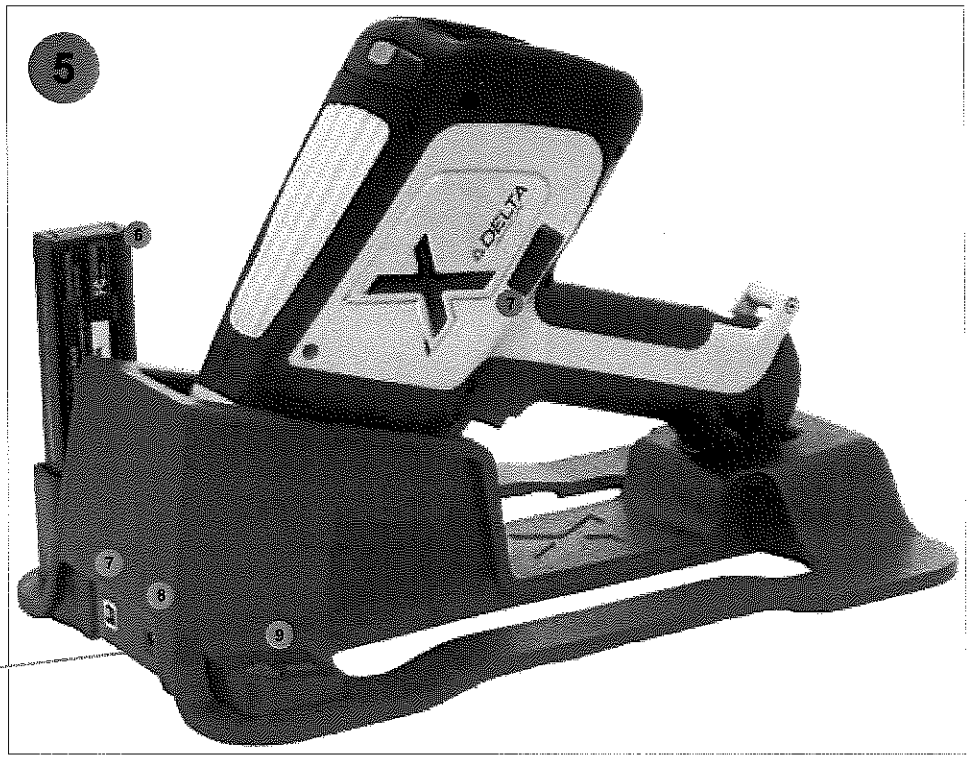
12 X-ray Warning Light Array

2. Delta Docking Station (DDS)

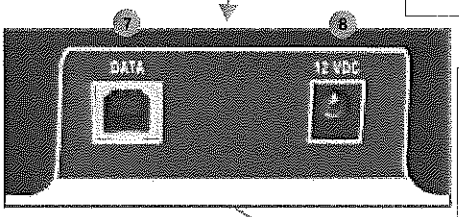


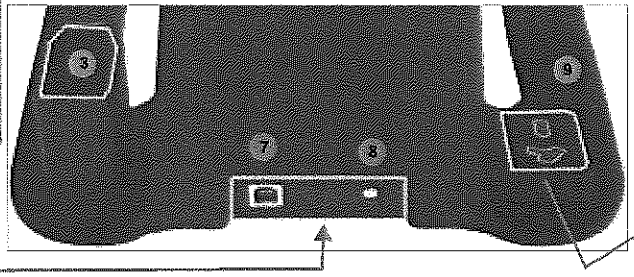
Component Key

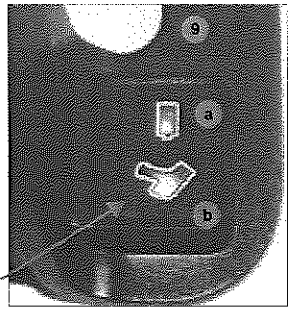
	Delta – All Model
1	Delta Docking Station (Empty)
2	Analyzer Signal/Control Connector
3	Spare Battery Charge Socket
4	CalCheck Test Cup (316 stainless steel coupon)
5	Docking Station (Loaded)
6	Second Battery in Socket
7	Data Port(s): — Docking Station –>Rear
	— Analyzer –> Left Side
8	Input Power (12 VDC)
9	Indicator Lights
a	Second Battery Charging
b	Analyzer Engaged



See C4/Pages 44-45 for more information







3. Accessories- List the Standard and Optional Accessories

Standard

- Batteries
- Delta Docking Station (DDS)
- DDS Power Adapter
- USB Cables 1 - USB mini to USB A
- USB Cable 2 - two part powered data cable
- Windows- Bags of Kapton and Prolene films
- Cal Check (Standardization) Coupon

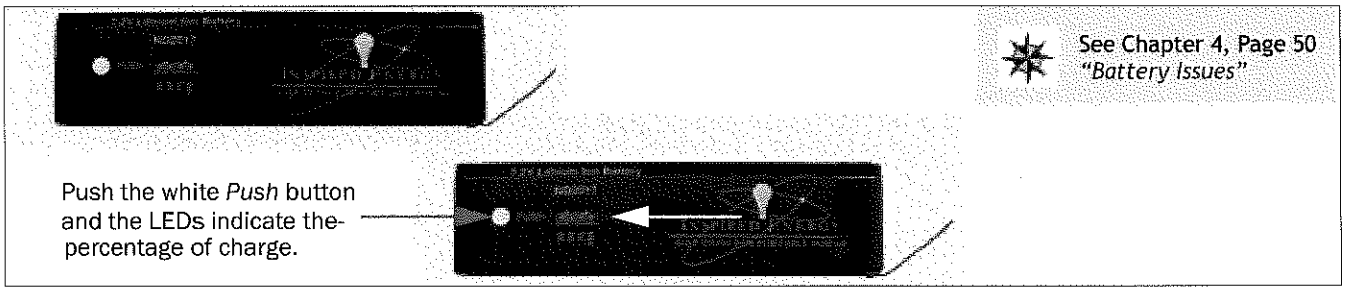
Optional

- AC Power Adapter (Battery Replacement)
- A-020-D TestStand/Workstation for Delta

STANDARD Accessories

Batteries

Two removable Li-ion batteries are standard accessories for the Delta.

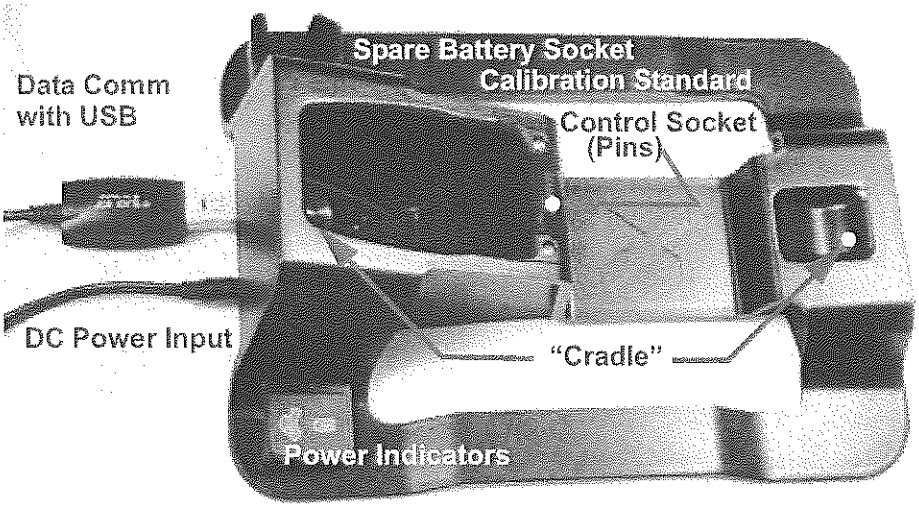


See Chapter 4, Page 50
"Battery Issues"

Delta Docking Station (DDS)

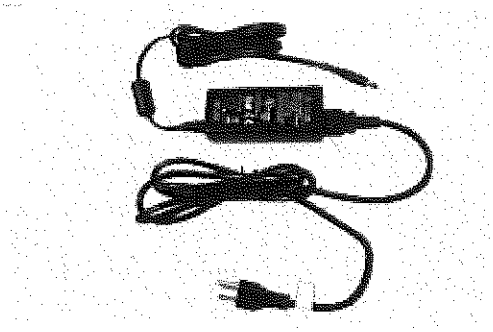
This is key accessory. It provides **three** functions:

- Cal Check by one of two means - "On Demand" or Automatically
- Charge internal battery in handle
- Charge additional battery in auxiliary socket



See Chapter 4, Page 46
"Use DDS for Start Up & Initial Cal Check"

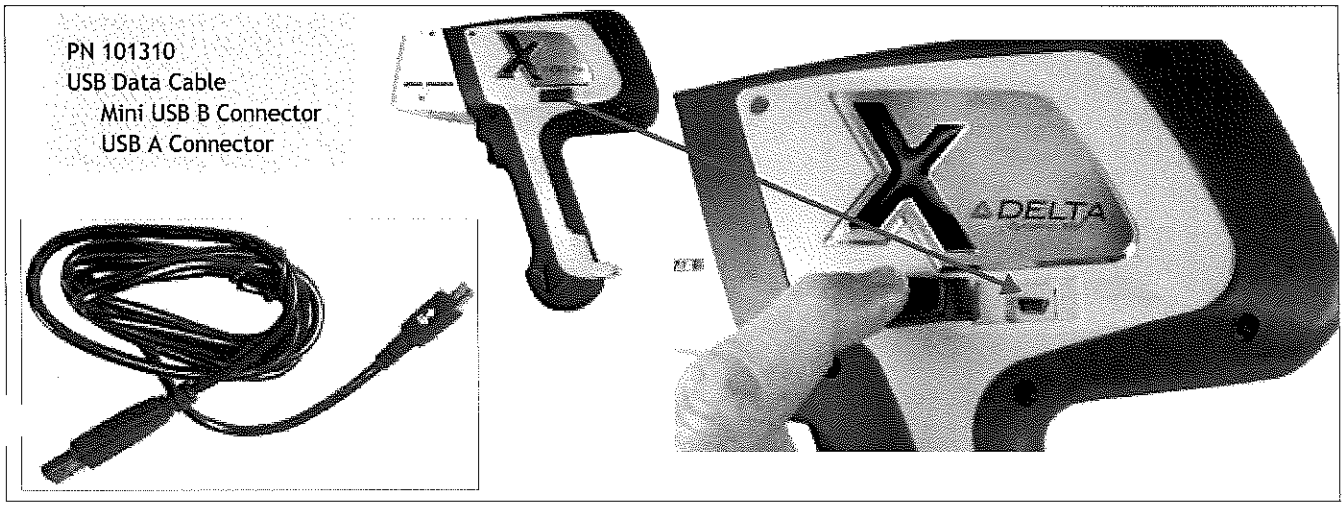
Power Adapter for DDS



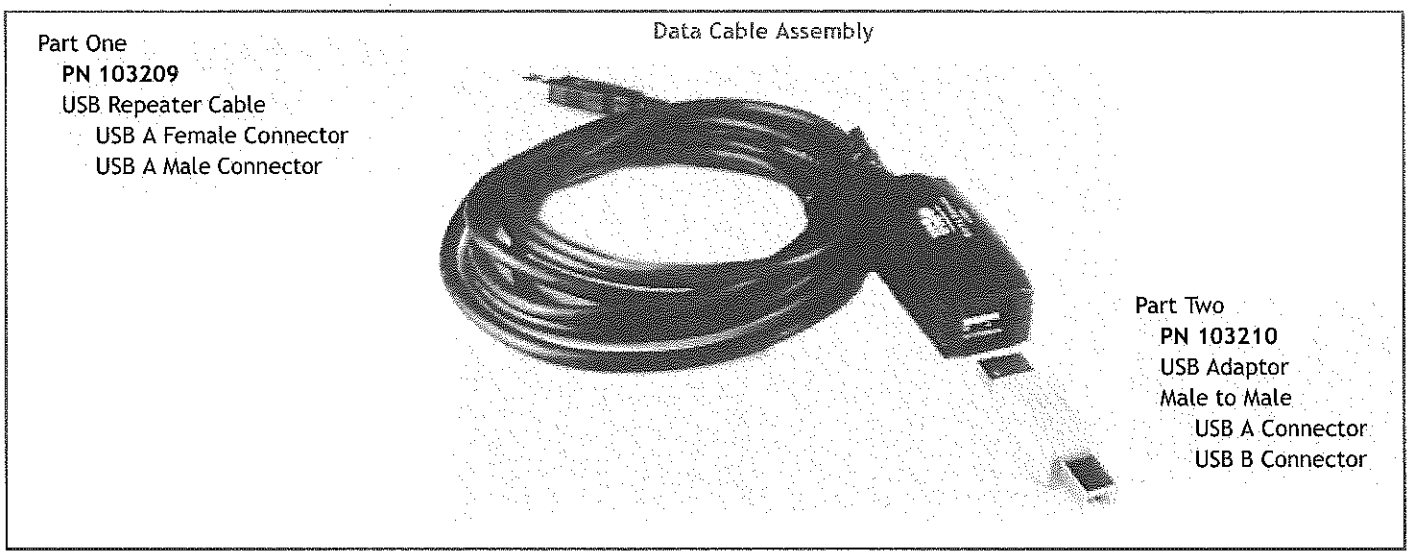
See Chapter 4, Page 44
"Configure DDS"

I/O Cables

PN 101310: This ***standard** accessory* provides a means to transfer information into or out of the sealed analyzer. It is good practice to export the current day's testing results to your PC.

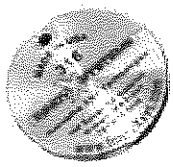


PN 103209 and 103210: This is a two part assembly that supports communication between the Delta Docking Station and a PC.



Cal Check (Standardization) Coupon

This part is used as a reference sample to provide a test standard for a Cal Check procedure if the Docking Stations is not available. The instrument indicates when a Cal_Check is necessary.

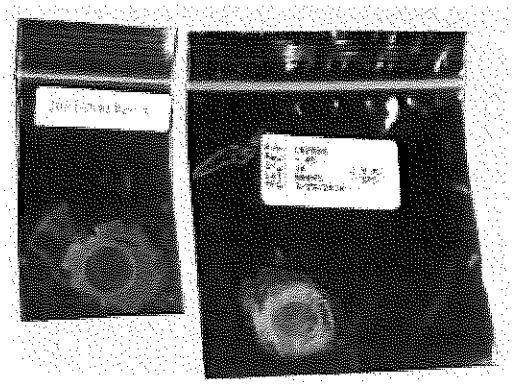


See Chapter 4, Page 51
"Cal Check Information"

Measurement Window Films

A bag of 10 window films are a standard accessory. The composition of the film is model and application dependent

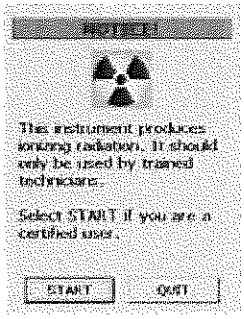
PN 103115 REV A
Kapton Windows
for Classic

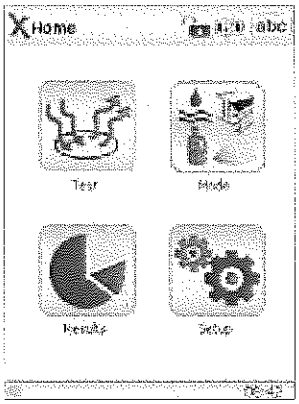


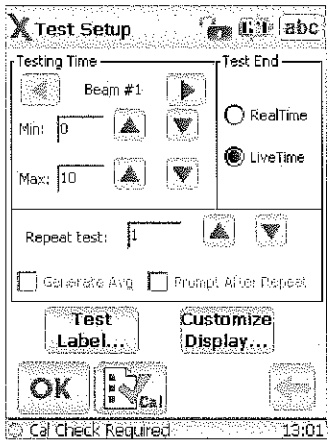
PN 102999
Prolene windows for
Standard and Premium

Application Software

The ***Delta*** instrument is shipped with proprietary InnovX data acquisition and processing software and Windows Embedded CE® operating system. The User Interface employs an icon-based home page graphic style. Factory calibration has been completed on all purchased modes.





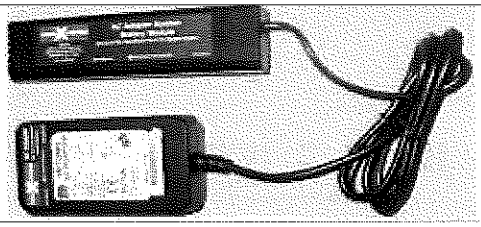


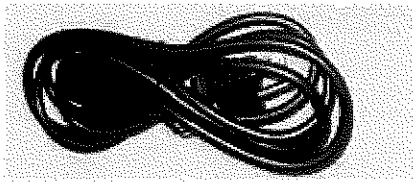
Sedately *User Interface Guide"* (PN 103202) for a complete description of the Innov-X application's User Interface.

OPTIONAL Accessories

AC Power Adapter

PN 100043: This accessory enables the user to operate the instrument without the limitation of battery charge status. The unit comes with approximately ten feet of power cord that defines the effective range of use.





PC Software

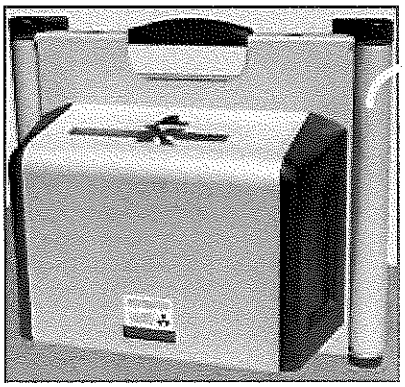
This application package permits an operator to execute Innovx S/W functions from a PC. With cable PN 101310, a user can connect from the Delta's mini-USB data port to a PC's USB port.
This package is optional for a handheld instrument and standard for A-020-D TestStand/Workstation.
When used with the A-020-D the proper configuration cable is the powered USB assembly (PN 103209 - PN 103210)

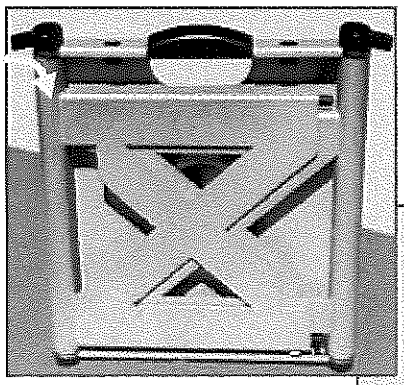
XRF Workstation

The Delta XRF Workstation is comprised of two major components:

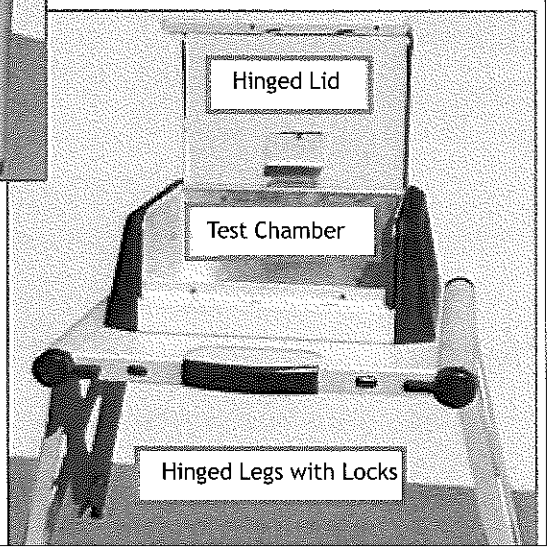
- A-020-D Test Stand, and
- Any Delta analyzer

In this configuration, the Delta is controlled by Innovx Delta PC Software. The open-beam handheld instrument is converted to a closed-beam workstation.





See PN 103158 document: *"HowTo-Setup & Configure Delta XRF Workstation"* for complete instructions.



The **XRF Workstation** offers the following features:

- Portable, light-weight, shielded enclosure
- A rugged and repeatable testing environment
- Easily erected in laboratory or at remote field site

In this configuration the Delta (an open-beam handheld instrument) is converted to a safe closed-beam system

C2. Safety Information

C2 presents the following information:

- Radiation Safety Information
- General Precautions
- Electrical Precautions
- X-Ray Safety
- Compliance
- Instrument Usage Scenarios
- Radiation Dosage for Several Scenarios
- Radiation Safety: Common Questions and Answers
- Analyzer Shut Down Procedure

Radiation Safety Information

Always make Operational Safety your HIGHEST PRIORITY.

The *Delta* Handheld XRF Analyzer is a secure and dependable instrument when used according to Innov-X's recommended testing techniques and safety procedures. However, this instrument produces ionizing radiation; only individuals trained in correct operating techniques and authorized to use X-ray producing devices should be permitted to use it. The radiation detected at any outside surface (excluding the Prolene, Mylar, or Kapton window area) is below that required for an unrestricted area.

- Heed all warning labels and messages
- Observe the safety interlock features

WARNING

X-ray tubes in Delta instruments can emit dangerous levels of ionizing radiation.

Prolonged exposure can cause serious illness, injury, or death.

It is the responsibility of Innov-X Systems' customers to follow the operating instructions and safety recommendations of this guide and good radiation control practices.

Radiation Safety Program

Innov-X strongly recommends that organizations using *Delta* analyzers implement a formal *Radiation Safety Program* that includes:

- Dose monitoring of critical personnel.
- Monitoring of area radiation levels.
- Information specific to the site and application of the XRF system.
- An annual review (and update, if necessary).

"C3. Safety Administration" provides a more comprehensive safety discussion for operators and managers.

X-Ray Safety

X-ray safety is a priority at any time and in any testing situation.

WARNING

- **Innov-X analyzers must be used by trained and authorized operators, according to proper safety procedures. Improper usage may circumvent safety protections and could potentially cause harm to the user.**
- **Heed all warning labels and messages.**
- **DO NOT USE the instrument if there is any chance that it is damaged or might leak radiation. In such a case, arrange for qualified personnel to perform a radiation safety test and repair any analyzer damage.**

General Precautions

Apply these general safety guidelines when managing or operating the *Delta* instrument:

- Retain and follow all product safety and operating instructions.
- Comply with all warnings on the product and in the operating instructions.

Comply with the precautions listed in this section to reduce the risk to:

- Users
  - Physical injury
  - Electric shock
  - Radiation exposure
- Equipment damage
  - Measurement window
  - Overheated electronics and other internal components

Service Considerations

Except as expressly noted here, do not service any Innov-X product yourself. Opening or removing the external housings may expose you to electric shock and the instrument to mechanical damage. It also voids the warranty.

CAUTION

If service is required, it must be performed by Innov-X or its authorized service representatives. Failure to observe this can result in loss of warranty. The **ONLY EXCEPTION** is replacing a damaged measurement window (see *"A5. Window Replacement"*).

Damage Requiring Service

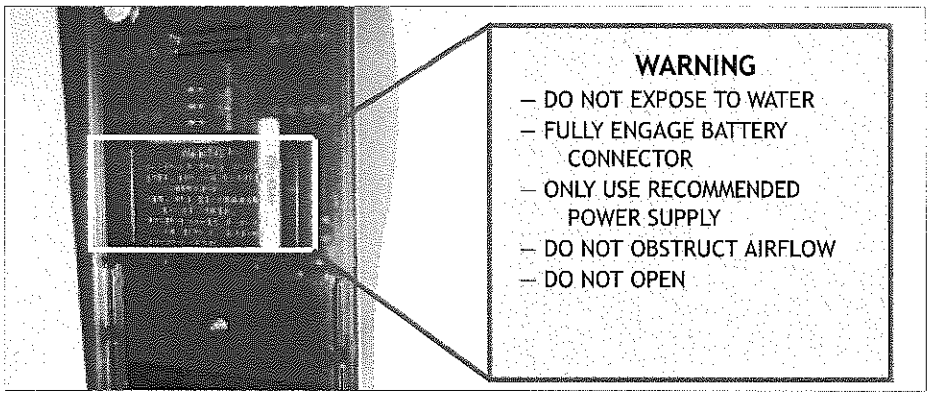
Types of problems or conditions that require service are (*but not limited to*):

- Power cords are damaged.
- Excessive or corrosive liquids spilled on the instrument or accessories.
- Instrument impacted, dropped, or physically damaged.
- Noticeable signs of overheating.
- Instrument or docking station does not perform normally when you follow the usual operating instructions.

Electrical Precautions

Guidelines for safe electrical operation of a *Delta instrument*:

- Use the correct battery or AC power adapter.
  - Install the battery or AC power adapter carefully, don't damage connections.
- Use the correct external AC power sources for the Delta Docking Station (DDS) (battery charging and Cal Checking) and the AC power adapter:
  - Ensure that the voltage is appropriate (100V-240 V/ 50-60 Hz) for operating either accessory.
    See *"A3. Specifications"* for electrical specifications.
- Do not overload an electrical outlet, power strip, or convenience receptacle.
- Do not exceed 80% of the branch circuit rating.
- Comply with the warning messages on the under side of the Battery Charger.
  - Similar precautions should be observed for the Delta Docking Station (DDS).



Cables and Cords

The *Delta* instrument and docking station is delivered with:

- AC power adapter (1) for Docking Station (standard)
- AC power adapter (2) as battery replacement for instrument (optional)

Each device has a standard IEC 3 conductor power cord which includes a safety grounding plug.

- If necessary, have an authorized individual replace these plugs to conform to local conventions.

Two data cables are supplied:

- Data cable (1) with connectors – USB A to USB B
- Data cable (2) with connectors – USB A to mini USB B

Cable Guidelines

Use these guidelines to ensure safety and proper equipment performance:

- The power cords MUST be connected to a properly grounded and easily accessible power outlet.
- Use a surge protector device, if possible.
- Do not defeat or bypass the ground conductor.
- Do not pull on cords or cables. Grasp the plug housing when removing the cord from the electrical outlet.
- Install all cords in accordance with applicable regulations.
- If you substitute a USB cable, ensure that the length doesn't exceed 10 feet.

Delta Docking Station (DDS) and Li ion Battery Packs

Plug the Delta Docking Station (and optional battery charger, if utilized) into a grounded electrical outlet that is easily accessible at all times.

- To handle battery packs properly do not:
  - Disassemble
  - Crush
  - Puncture
  - Short external contacts
  - Dispose of in fire or water
  - Expose to temperatures higher than 60 °C (140 °F).

GO TO

See *"C4. Battery Issues"* for instructions concerning Batteries, the Battery Charger, and the AC Power Adapter.

WARNING

Danger of explosion if battery is incorrectly substituted.
Replace only with Innov-X specified batteries.

Used batteries may be returned to Innov-X Systems for disposal.
If returning batteries, or equipment with batteries installed, the shipping container must display a special caution label.
See *"A6. Packing and Shipping"* for label details.

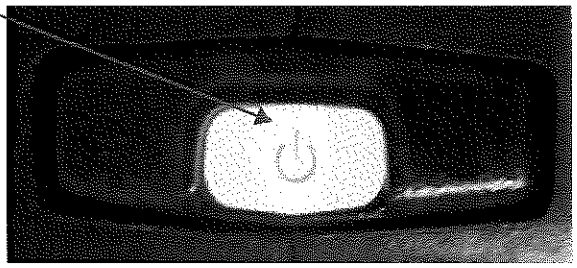
Indicator and Warning Lights

Power Switch w/ Integral Indicator Light

The Delta power switch is located at the upper rear of the unit.

POWER ON

- Press the I/O switch to turn on the power.
  - A green LED indicator comes on.
- This switch ***DOES NOT*** turn on the x-ray tube.
  - No tube power supplied until the Innov-X software is launched and a test is initiated.

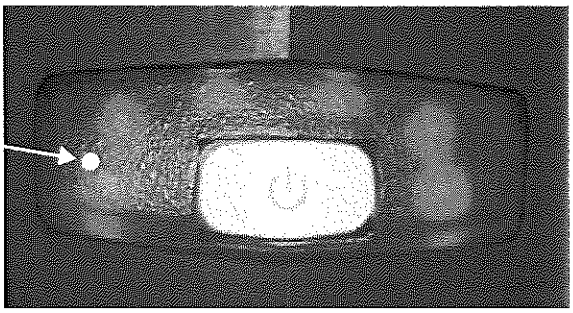


POWER OFF

- Press and hold switch for >3 seconds.
  - Unit powers off. (See page 36 for more Exit options)

X-Ray Indicator Light Array

An indicator light array (six red LEDs) alerts the operator when the tube is receiving power, and when x-rays are emitted from the analyzer through the measurement window.



- When the unit is initially powered ON, the Indicator array remains Off.
- As test is conducted, array is in a **flashing** state.
- At the test's conclusion, the array stays **on continuously** until the beginning of the next test.

X-Ray Indicator ON (*Blinking*)

When the indicator array is flashing, this signifies:

- X-ray tube is powered to full operational level
- Internal filter wheel is in operational position
- Analyzer is emitting x-ray radiation through the analysis window.

In this condition, the analyzer must be pointed at a test sample.

X-Ray Indicator ON Continuously (*Not Blinking*)

When the indicator array is on continuously, this signifies:

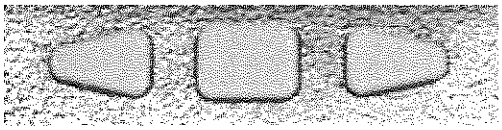
- X-ray tube's current is set to 0.0
- X-ray tube is producing a minimum level of x-rays
- Internal filter wheel is closed so there is no radiation exposure to you or bystanders.

The instrument is ***safe*** to be carried or set down in this condition.

Back of Analyzer

In addition to the I/O switch and the X-Ray indicator array, the back of the Delta analyzer has:

- **Touch screen** which displays and controls the Delta User Interface.
- **Three Navigation Buttons** below the screen. They permit the user to conveniently step through the Test Results Spectrum screens.



Navigation Buttons

X-Ray Label

The Delta has a warning label affixed to the lower surface of the probe.

The analyzer has a label on the lower surface of analyzer's probe/nose.

- This label is ***required*** by most regulatory agencies. Do not remove it.
- The label term "*WHEN ENERGIZED*" refers to the condition where the tube is fully energized and the filter wheel is open.
  - This condition corresponds with the blinking red LEDs that comprise the X-ray indicator array.

Other Safety Features

Proximity Sensor

The Delta automatically detects when it is engaged with a test sample.
It immediately shuts off the X-ray tube if:

(a) Initially there is no sample in front of the window,

— or —

(b) Instrument is pulled away from the sample before the test time has expired.

— Back of Analyzer —

Safety Interlock Structure

For controlling the Delta's X-ray emissions and therefore minimizing the possibility of accidental exposure, there is a standard safety interlock structure consisting of the three features listed below.

Software Trigger Lock

- If five minutes elapse between tests (default time), the trigger locks automatically and you must tap on the lock icon to unlock it. See Safety Software instructions in *"Delta User Interface Guide"*.

Software Proximity Sensor

- Within two seconds of a test start, the analyzer detects a sample in front of the measurement window. If not, the test aborts, the filter wheel closes, and the x-rays shut off. The tube is placed in standby and the red light stops blinking.

Safeguards

As an owner of an Delta handheld XRF instrument, your safeguards are:

A. Limited Access
B. Trained Operators
C. Shielding Issues

A. Limited Access

Keep the instrument in a controlled location, where only trained and authorized users are likely to have access.

B. Trained Operators

Keep a sign with the analyzer indicating that in order to use it an operator must have completed a training class provided by your company, or must have attended an Innov-X training course and completed any other requirements as dictated by the local regulating authority. When the Innov-X system is turned on, the controller screen displays a message indicating that the system should only be used by authorized personnel.

C. Shielding Issues

Background

The Delta emits a tightly collimated beam of X-ray radiation. The beam projects many meters when only air attenuates it.

NOTE

Refer to governing regulations on compliance in the jurisdiction installed, dose limits, etc. Requirements differ from state to state, region to region, country to country. *DO NOT* rely solely on this manual for instruction.

Action

Adequate shielding is achieved by:

- Establishing a *no-admittance zone* sufficiently distant from the instrument's measurement window that allows air to attenuate the beam.
- Enclosing the beam working area with *protective panels* (for example, 1/8" stainless steel can attenuate the beam to background levels)

Contact your Innov-X Systems representative for assistance and suggestions on interlocks and applications for limiting radiation exposure.

Instrument Usage Scenarios

The Delta is used in several testing configurations. Obey the guidelines listed below.

Practical Safety Guidelines for Handheld Analyzers

WARNING

- **DO NOT POINT the unit at yourself or any other person during operation.**
- **Never perform a test by holding the sample with your fingers or in the palm of your hand.**
- **Always wear both a ring-style and a badge-style dosimeter.**

Correct Usage

Test in Place

Test targets can include pipes, valves, large pieces of scrap metal, soil, or any sample large enough to be tested in place.

In this configuration the proper procedure is as follows:

1. Always observe the relevant parts of the *Practical Safety Guidelines* shown above.
2. Point the instrument at the sample such that *no part* of your body (including hands and/or fingers) is near the measurement window.
3. Ensure that the Delta's nose (with window) is firmly placed on the target.
4. Perform the test using one of these methods:
   - Tap **Start** on the UI

   — or —
   - Pull the trigger (this toggles the instrument to ON state)

   — or —
   - Pull-and-hold the trigger with the "deadman trigger" active.

Employing Steps 3 & 4 assures that no operator's body part is exposed to an excess radiation dose. The radiation detected at user interface areas is < 5 μSv/h.

Take care that during testing, personnel are not located within three feet (one meter) of the Delta's probe head, in the direction of the x-ray beam. Provided the window is completely covered, there is minimal radiation being emitted around the area of the sample.

Small Component Testing

Examples of small component targets include metal turnings, weld rod, wires, fasteners, nuts and/or bolts.

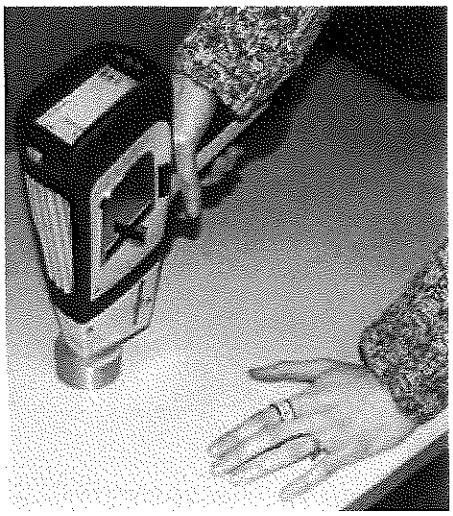
For analysis of these types of components, use this procedure:

1. Always observe the relevant parts of the *Practical Safety Guidelines* shown above.
2. Place the sample on a flat surface.
3. Carefully place the nose/window over the sample.
4. Perform the test using one of these techniques:
   - Tap **Start** on the UI

   — or —
   - Pull the trigger (this toggles the instrument to ON state)

   — or —
   - Pull-and-hold the trigger with the "deadman trigger" active.

1. Sample lying on a flat surface



SAFETY PRECAUTIONS

WARNING

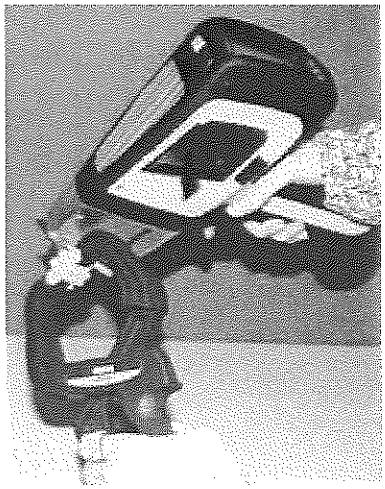
Do not test samples while sitting at a desk or table If the desk is made of wood or another non-metallic material, some radiation will penetrate the desk and may provide exposure to legs or feet.

ANALYTICAL PRECAUTIONS

NOTE

If the sample does not completely cover the window, ensure that your *background surface* does not contain metals or even trace levels of metals, as this may affect the accuracy of the XRF result. The XRF may report the presence of additional metals in the surface material.

2. Clamp-held sample



NOTE

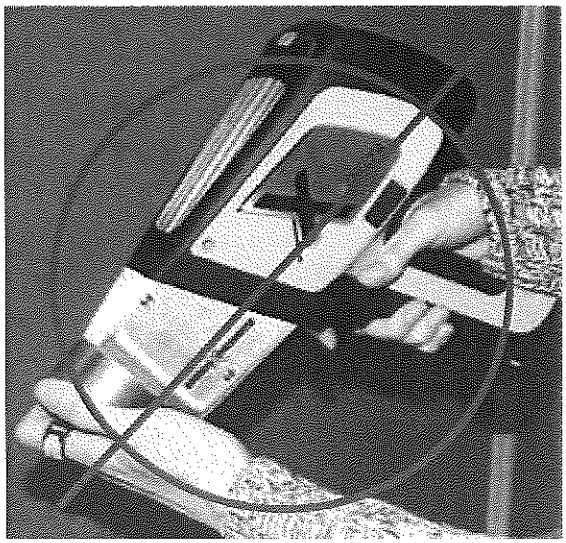
A handheld plastic locking clamp can be an effective and safe tool when analyzing small, irregular shaped samples.

Incorrect (Unsafe) Usage

WARNING

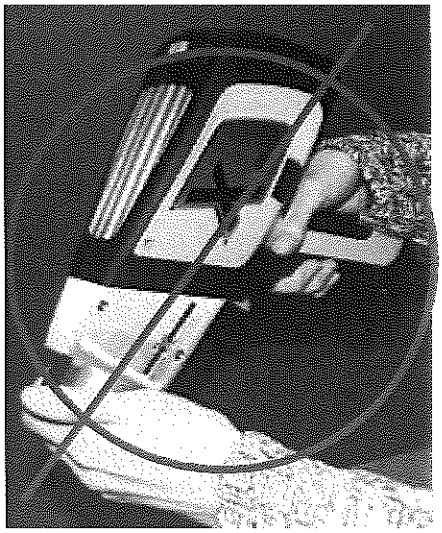
Never hold a sample in your hand such that any part of your body or appendages are exposed to the x-ray beam. Testing samples in this way may generate significant radiation exposure to your fingers.

Unsafe Testing Technique



The sample is held up to the measurement window with fingers. The sample does not completely cover the window.
Even though the analyst is wearing a ring dosimeter, this is an unsafe testing technique.
Here, the only value that the ring provides is to validate the level of unnecessary radiation exposure that has been experienced.

Unsafe Testing Technique



The sample is held up to the measurement window with fingers. The sample does not completely cover the window.
To compound the danger, the analyst is not wearing a ring dosimeter.
There is no measure of the radiation exposure endured.

Summary

InnovX repeats the Warning ---

NEVER hold a sample in your hand.

Testing samples in this way generates significant radiation exposure to your fingers.

Compliance

Complying Agency Statements

United States of America: FCC

Changes or modifications not expressly approved by Innov-X Systems, Inc. could void the user's authority to operate the equipment.

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to Part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications.

Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

Radiation Doses for Several Scenarios

In this section we provide data, concrete examples of use and misuse of the analyzer and common questions and answers we encounter when training personnel on the safe use of the Innov-X analyzer. The goal is to explain scenarios of safe versus improper usage.

WARNING

For the x-ray energy emitted by portable XRF analyzers (8-60 keV region), the bone in the fingers will absorb radiation about 3-5 times more than soft tissue, so the bone would be at an elevated radiation risk compared to soft tissue.
For this reason, no person shall hold a test specimen in front of the window with the fingers in the direct beam, or direct the beam at any part of the human body.

Reference: *Health Physics 66(4):463-471;1994.*

The table below presents radiation doses for normal operating conditions and also for examples of misuse of the analyzer and even extreme misuse. Innov-X provides installation training that includes detailed radiation safety training and documentation designed to prevent misuse of the analyzer.

Although the doses shown below are derived from experiments with TLD (thermo-luminescent dosimeters) and may or may not represent actual absorbed dose in human tissue and bone in each scenario, they are examples of the level of x-ray radiation being emitted from the device.

The message is simple:

USE CAUTION AND PROPER TECHNIQUE when operating the device.

Example: Instrument Usage	Radiation Exposure and Comments
Normal Operation- Dose to Hand User analyzes samples according to standard operating procedures described in this manual. Assumption: Operator using system with x-ray tube ON for eight hours/day, five days/week, 50 weeks/year. (Alloy sample).	Maximum exposure is to operator's hand, at the trigger is < 1μSv/h. Annual exposure to hand is then < 2mSv. Maximum exposure under ICRP regulations is 500 mSv for radiation workers and 50 mSv for the general public. Thus continuous operation provides a dosage 250 times lower for a radiation worker and and 25 times lower for the general public.
Normal Operation- Dose to Torso Analyzer is used under the same operating conditions described above.	Exposure to Torso is so low it cannot be measured (essentially background). To be conservative we use 1/2 the value as the trigger, < 0.5μSv/h. Annual exposure using operating conditions above is then estimated at less than 1 mSv. Maximum allowed is 20 mSv under ICRP for radiation workers (1 mSv for general public).
Misuse Example 1: Operator holds samples in front of window with fingers, such that fingers are directly in the primary beam. Presumption is sample does not block any radiation. Do not do this!	At the window, in the primary beam, the maximum dose to the fingers is 20,000 mSv/hr. Assume an operator performs a 10 sec. test (typical). The dose to the operator's fingers or hand is 20,000 x (10/3600) = 55 mSv. If the operator did this just ten times per year he would exceed the allowable annual dose of 500 mSv to an extremity. Take the extra time to test a sample on a surface or use a testing stand. **Note: If the operator takes a shortcut and places his/her fingers within the primary x-ray beam at the window, they will exceed the annual dose rate.**
Misuse Example 2: Operator places analyzer against body and pulls the trigger to start a test. Analyzer tests to preset testing time (usually ten seconds) unless operator pulls trigger again to stop test. This applies to analyzer being in contact with operator or with bystander. Do not do this!	Dose at exit of sampling window is 20,000 mSv/h. Dose for a ten second exposure with analyzer in contact with Torso: 55 mSv. If an operator did this act just once, he would exceed the annual safe dosage to the torso of 20 mSv/year by a significant amount! **PLEASE NOTE:** The maximum dose of 20 mSv/year is a whole body limit, which does not truly apply in this case because the x-ray beam size is small (about 25 mm^2 area at the port). Applying correction factors for the beam size is complex and beyond the scope of this manual. The important point is that for proper operation there is no reason to ever expose any part of the human body directly to the x-ray source. This example serves to provide estimated exposure in the event this occurs.

Misuse Example 3: Operator manages to initiate a test for ten seconds running normal soil mode and exposes a bystander that is standing ten cm away from analyzer port. What is exposure to bystander? **Note:** The **proximity sensor** would automatically shut down the x-ray tube immediately, so this is an extremely improbable occurrence. It would require a malfunction of the instrument - this safety feature in NOT modifiable. **Note 2:** Equations to scale these to other scenarios involving longer or shorter tests, and bystander being at distances other than ten cm are provided at right.	Dose to bystander at ten cm is 215 mSv/hr. For a ten second exposure the dose is 0.6 mSv. This is 33 times lower than the allowable dose to a nuclear worker in a year. This would have to happen 33 times to for that worker or bystander to obtain the maximum allowable dose. Formula for calculating other scenarios: DOSE (in mSv) = $6T/D^2$ D = distance from port in inches T = testing time Example: Bystander is 30 cm away from port for a 30 second test. In this case the dose is calculated as: DOSE = $6(30)/30^2$ = 0.2 mSv

Comparative Analysis:

Radiation Doses from Typical Exposures to Ionizing Radiation

Activity	Typical Dose
Smoking	***2.8 mSv per year***
Dental x-ray	***100 μSv per x-ray***
Chest x-ray	***80 μSv per x-ray***
Drinking water	***50 μSv per year***
Cross country round-trip by air	***50 μSv per trip***
Mammogram	***1-2 mSv per examination***
Yearly exposure from background* radiation * depends on geographic location	***3.6 mSv***

Radiation Safety: Common Questions & Answers

Question: When I'm shooting a piece of pipe or valve on a rack or on a table top, is there any exposure to people standing several feet away from the analyzer?

Answer: Even a thin amount of a dense metal sample (three to four mm thickness, not Al alloy) is enough to completely attenuate the emitted x-ray beam. Shooting a piece of material that covers the sampling window on the analyzer completely shields any bystanders from radiation exposure.

However, use good practice: Keep the area clear of people for at least four to five feet in front of the analyzer.

Question: If I forget to lock the trigger, I pick up the analyzer and accidentally pull the trigger, is that dangerous to nearby personnel?

Answer: No, this example of misuse is not dangerous, but it may produce a non-negligible radiation exposure to nearby personnel. For an exposure to occur, the following things must happen.

First, you must be holding the analyzer so that a bystander is actually standing in the x-ray beam being emitted. Just being near the analyzer is totally safe otherwise.

Second, the bystander must be within one meter from the nose of the analyzer to receive any appreciable dose. If all of these conditions are true, the dose received by a bystander is still extremely low. Please see Misuse Example 3 in the table above.

Third, it would require failure of the proximity hardware and software.

Question: Do I need to create restricted areas where I am using the analyzer?

Answer: No, provided you are following normal operating procedures there is no reason to restrict access to an area where the analyzer is in use. However, the operator should take precautions to keep any personnel more than three feet away from the sampling window of the analyzer in the event of accidental misuse as detailed above. Should operators also elect to test small samples as shown on pages 34 and 35, they should also be sure that no personnel are standing within about four to five feet of the sampling window.

Question: How does the x-ray tube in the Innov-X system compare to a radiography system used for taking images of metal parts?

Answer: The x-ray tube used in the Innov-X system produces between 1,000 and 10,000 times less power than most radiography systems (0.5-1 watt versus multiple-kW). A portable XRF is designed to perform surface analysis of alloys and other samples, whereas a radiography system is designed to shoot x-rays entirely through metal components in order to obtain an image on the other side of the test object. For example, many tube-based radiography systems use a 300-400 kV tube and currents in the tens or hundreds of milliamperes (mA). The Delta uses a tube operating at a maximum of 40kV and typically 6 -10 μA.

The radiation levels produced by an Delta are thousands, or tens of thousands, times lower than a radiography unit.

Question: Should we use dosimeter badges with the Innov-X analyzer?

Answer: Dosimeter badges are required by some provincial regulatory agencies, and optional with others. Innov-X recommends that operators wear badges, at least for the first year of operation, as a general precaution to flag any misuse of the analyzer. Dosimeter badges are available for the torso (generally worn in a shirt pocket) and also as "ring" badges.

The best practice is to wear a ring badge on a finger on the opposite hand used to hold the analyzer. This records accidental exposure for the most likely case - an operator grabbing a small sample and holding it in one hand while analyzing it.

Note: These badges generally have a threshold of 100 μSv and are renewed monthly. So it takes several cases of misuse even to obtain a reading on a typical badge. When purchasing a badge, obtain the type used for x-ray and low energy gamma ray radiation.

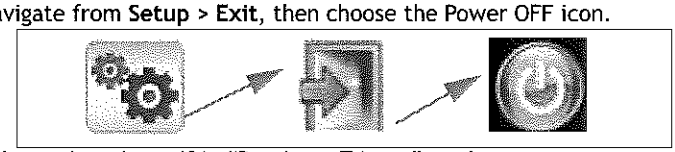
Analyzer Shut Down

There are several techniques for shutting off the Delta. They can be categorized by whether the action is taken under normal or emergency conditions. *Shut down* or *turned off* is defined as: *The analyzer cannot provide X-ray emissions*.

Under normal conditions

Use one of following actions:

- Press the trigger.
- Tap **STOP** icon on the UI touchscreen.
- Navigate from **Setup** > **Exit**, then choose the Power OFF icon.



- Release the trigger if in "Deadman Trigger" mode.
- Press the I/O power switch; ensure that the On/Off LED goes off.

In an emergency

Because the Innov-X system is a battery-operated, x-ray tube-based analyzer, the Emergency Response plan is simple. If you believe that the analyzer is locked up in an *OPEN* position, the red X-ray indicator array remains illuminated or blinking:

1. Press the I/O power switch as noted above. If the power does not turn off, continue to Step 2.
2. Open the battery cover and immediately remove the battery.

If you are using the AC Power Adapter:

- Remove the Battery Eliminator from the Delta's handle

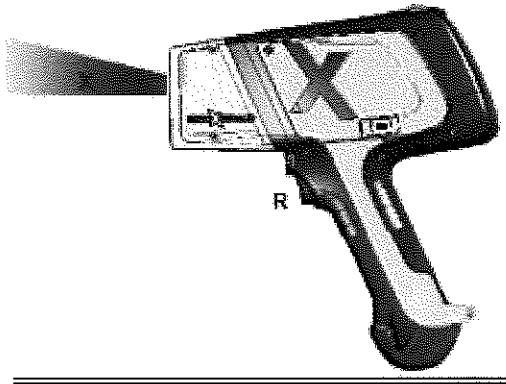
— or —

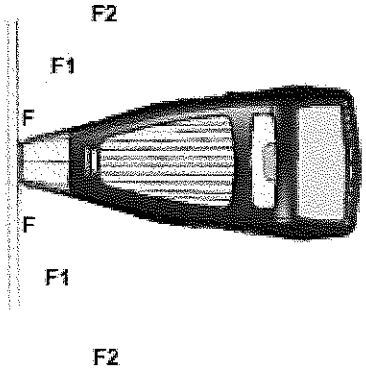
- Pull the AC cord from the AC Power Adapter or pull the plug from the receptacle.

Delta Radiation Profile

This is the current Delta Radiation Profile.

RADIATION PROFILE – Hand Held Instruments





Measured Shallow Dose Rate in mR/h - Secondary Radiation (Scatter)

Mode(s)	Substrate	Voltage kV	Amperage µA	Filter	Trigger R	Close F	10 cm F1	30 cm F2
Alloy Plus Mining 2-Beam Mining	316 Stainless	40	100	1	0.1	1	0.5	0.1
	Al (319 AA)				0.2	5	3	0.5
	Soil (SiO_2)				3	50	25	8
Alloy Plus 2	316 Stainless	13	200	7	BK	0.1	BK	BK
	Al (319 AA)				BK	BK	BK	BK
2-Beam Mining 2	Soil (SiO_2)	10	200	7	BK	0.1	BK	BK
3-Beam Soil 1		40	100	3	1	15	7	2
3-Beam Soil 2		40	100	2	3	100	25	45
3-Beam Soil 3		15	200	5	BK	0.1	BK	BK

BK – No higher than background

TEST CONDITION: Instrument run at normal setting for mode and represents typical production unit.

NOTES

C3. Safety Administration

C3 provides information regarding:

- Radiation safety training recommendations
- Dosimeter badges
- A typical dosimeter monitoring program
- Dosimeter service contractors
- Typical registration requirements for operating XRF equipment (in the USA)

Radiation Safety Training Recommendations

Individual companies and states have specific regulations and guidelines for using ionizing radiation generated by an X-ray tube.

NOTES

For the convenience of clients, Innov-X has compiled a list of recommendations that:

- Provide generic guidance for an ALARA (*as low as reasonably achievable)* approach to radiation safety.
- Do not replace the requirement to understand and comply with specific policies of any state or organization.

Personal Monitoring

Radiation control regulations may require implementation of a radiation monitoring program, where each instrument operator wears a film badge or TLD detector for an initial period of one year to establish a baseline exposure record. Continuing radiation monitoring after this period is recommended, but may be discontinued if accepted by radiation control regulators. See *Dosimeter Suppliers* for a list of film badges providers.

Proper Usage

Never point the instrument at a person. Never point the instrument into the air and perform a test. Never hold a sample in your hand during a test.

Establish Controlled Areas

Restrict access to the location of instrument storage and use to limit potential exposure to ionizing radiation. In use, the target should not be hand held and the area at least three paces beyond the target should be unoccupied.

Specific Controls

When not in use, store the instrument in a locked case or locked cabinet.
When in use, keep it in the direct control of a factory trained, certified operator.

Time - Distance - Shielding Policies

Operators should minimize the time around the energized instrument, maximize the distance from the instrument window, and shoot into high density materials whenever possible.

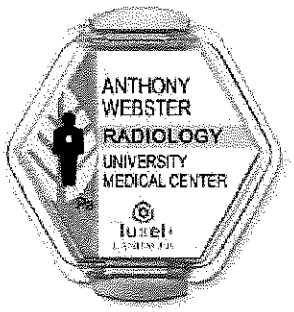
Prevent Exposure to Ionizing Radiation

All reasonable measures, including labeling, operator training and certification, and the concepts of time, distance, & shielding, should be implemented to limit radiation exposure to *as low as reasonably achievable* (ALARA).

Dosimeter Badges

A dosimeter badge consists of a radiation-sensitive material, generally an aluminum oxide crystalline layer, which is worn in a small container. It is most often attached to a person's clothing, on a belt loop, or shirt pocket. It is worn on the body in location(s) that most closely simulate the pattern of potentially absorbed dose.

The protection can also be provided in a plastic ring format. Here the detection material is lithium fluoride crystal.



Dosimeter
Clip-on Style

Dosimeter
Ring Style

These devices record a person's accumulated radiation exposure over a period of time. They monitor individuals working with, or near someone working with devices which emit ionizing radiation.

Dosimeter badges are required by some regulatory agencies, and are optional with others.

Innov-X recommends that (at a minimum) all *Delta* operators wear badges (both clip-on and ring styles) for the first year that their system is in use.

NOTE

- When purchasing a badge or ring, always select the type used for *X-ray* and *low energy gamma* radiation.
- Innov-X suggests that the ring badge be worn on a finger of the *opposite hand* used to hold the analyzer. This records accidental exposure for the most likely case: An operator grabbing a small sample and holding it in a hand while analyzing it.

Every country (including every region, state, or province within a country) can have differing regulations. Always consult your local radiation protection authority or Innov-X Systems for information and recommendations.

Dosimeter Safety Program

A typical dosimeter-based safety program uses the following steps:

1. The company develops a dosimeter program with an independent service contractor.
   - They establish the quantity of badges needed and the frequency of analysis (a monthly or quarterly interval)
2. The company receives the first lot of badges and provides them to their analyst/operators.
3. At the end of the interval:
   - The company collects the badges and returns them to the service contractor for analysis.
   - Simultaneously, the service contractor delivers another lot.
4. The company provides the new set of badges to maintain a continuous protection /monitoring program for their employees.
5. The service contractor prepares a report for the company that tabulates any X-ray dose received and identifies any personnel with readings higher than typical background radiation.
6. The safety monitoring cycle repeats with Steps 1 through 5.

NOTE

The service contractor's written records are very important to a company's overall safety documentation plan.

Dosimeter Suppliers

Some dosimeter service companies are:

Company	Location	Telephone
AEIL	Houston, TX	713-790-9719
Global Dosimetry Solutions	Irvine, CA	800-251-3331
Landauer	Glenwood, Il	708-755-7000
• Landauer, Inc.	Oxford, England	+44-1 86-537-3008
• Nagase Landauer, ltd.	Japan	+81-3-36 66-4300
• LCIE Landauer	Paris, France	+33-(0)1-40 95 62 90
• Landauer	Beijing, China	+86-10-62 21 56 35

Registration Requirements

Contact Innov-X for assistance with locating registration requirements information.

- Most states require some form of registration. Generally they require the registration to be received within 30 days of receipt of the system.
- Some states require no registration.
- Some states require notification in advance.

Customers are advised to consult their local radiation protection authority for specific regulatory information.

Typical Device Registration Information

The following information is usually requested by a licensing agency:

Purpose of device:

Response is *Analytical or* ***Industrial***.

Be sure to inform the government registration office that the system will NOT be used for radiography or for medical uses.

Radiation Safety Officer:

List person who monitors training, safe use, and controls access to the system.

Authorized Users:

List the analyst/operators who have been trained and authorized by the instrument owner and/or regulating agency to operate the XRF equipment.

Operating parameters of the Delta XRF analyzer:

8– 40 kV, 5 - 200 uA max.

Type of system:

Response is: ***Handheld/Portable***

User Training Specified:

Indicate that only individuals receiving manufacturer training, ***documented by a manufacturer's training certificate***, will operate the system. Additional training may be required. Verify with the local regulating agencies the level and type of training required.

Personal Monitoring

Many government agency registration forms require that you indicate whether or not you intend to perform dosimeter monitoring.

See "Dosimeter Safety Program" on previous page for information regarding typical personal radiation monitoring.

CAUTION

Always keep the following documentation at the job site:

- Copy of *License Registration*
- Other pertinent *government agency* documentation
- Copies of any *dosimeter analysis* reports
- Copy of this equipment's *User Manual*.

C4. Operations

This chapter provides information regarding:

- Configure the Delta Docking Station (DDS)
- Use DDS for Initial Cal Check
- Operation - General
- Start Up Procedure
- Snapshot of Delta User Interface
- Typical Test Procedure
- Ending Test Operations
- Battery Issues
- Additional Cal Check Information
- TIPS - Things You Should Know About the Delta

Safety First !

As emphasized in *"C2.Safety Information,"* it is a priority to keep the analyzer operator's safety in mind at all times.

- Operators, ***before turning on the analyzer or using the Delta Docking Station,*** should review the safety procedures (*"C2.Safety Information"*).

Set Up and Use the Delta Docking Station

Background

The Delta Docking Station (DDS) provides several key functions:

- Supports an automatic or on-demand Cal_Check procedure
- Charges the "Main" battery located in the instrument's handle
- Simultaneously charges a "Spare" battery in an auxiliary socket
- Provides control information so that both batterys' status can be monitored
- Allows data communication from the Delta to a PC via a powered USB cable

The first phase for preparing to operate a Delta involves:

1. Configuring the DDS with its power and communication cables
2. Using the DDS to support the Delta's initial:
   a. Start up sequence, and
   b. Cal Check procedure.

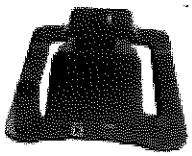
NOTE

A new instrument is shipped with two fully charged Li Ion batteries. Therefore, prior to **initially** using the analyzer, it is not necessary to charge a battery.

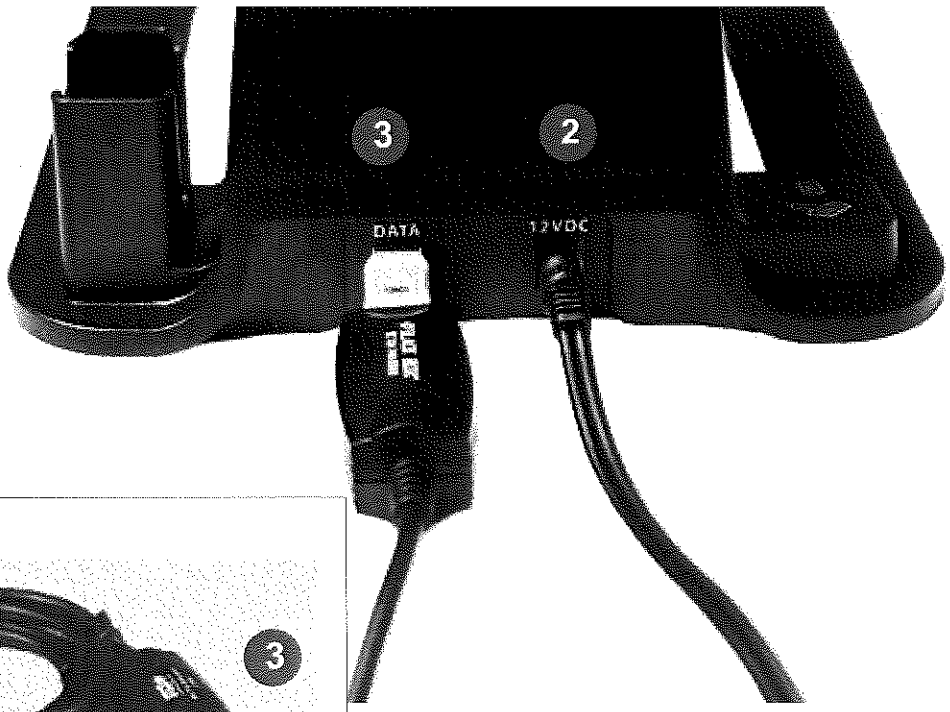
GO TO

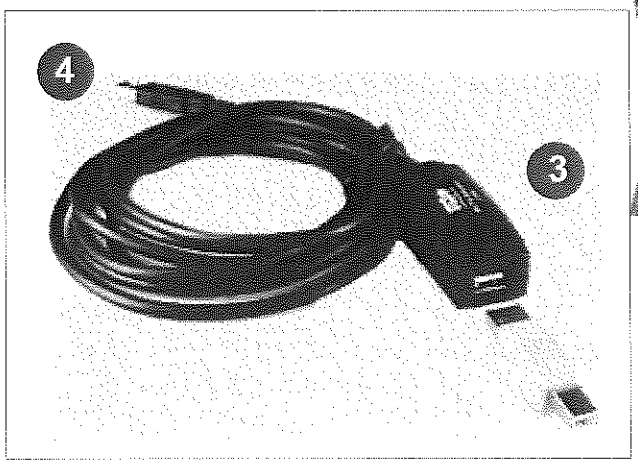
- See *Pages 45 and 50* for battery information, including charging, changing, determining status, and Hot Swap techniques.
- See *Page 51* for Cal Check background information.

Configure Delta Docking Station

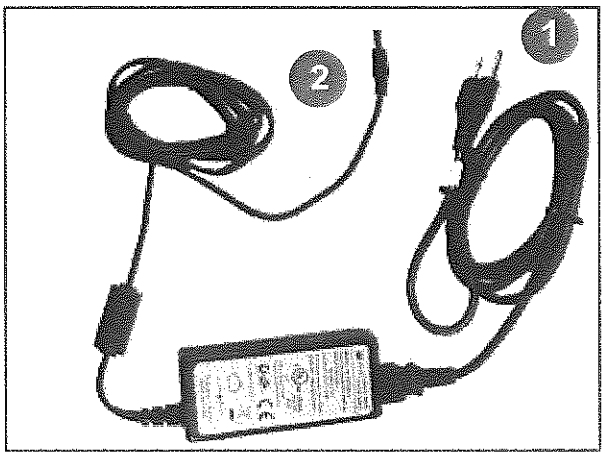


Rear View - DDS





PN 103209 & PN 103210
Powered Hub USB Cable Assembly



Power Adapter for DDS

Configure DDS with this procedure:

1. Plug the AC power adapter's line cord (1) into suitable AC outlet.
2. Plug the AC power adapter's DC jack (2) into the 12 Vdc socket on the rear of DDS
3. {Option at this point: Powered USB cable assembly}
   – Insert connector B (3) into DATA port
   – Insert connector A (4) into USB port of PC

Use the Delta Docking Station for Charging Batteries

The Delta analyzer has a multi-purpose standard accessory:
the **Delta Docking Station** or **DDS**.

In addition to providing an automatic Calibration Check, the DDS delivers **two** charging functions:

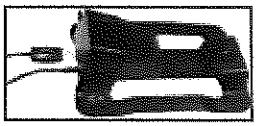
1. It charges the installed "Main" battery in the instrument's **handle**.
2. Simultaneously, it charges a spare or "Dock" battery when its inserted into the special auxiliary charging **socket**.

Charge status is shown in real-time on the Delta's display screen.

The second docked battery's status is also shown by the battery icon located on the rear left side of the DDS.

(either "charging = red" or "full = green")

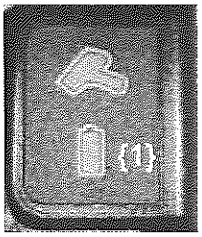
Battery Status Screen
(Delta inserted in DDS)

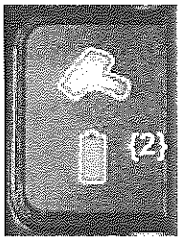


Cradle

Delta Docking Station Procedure for
Battery Charging Functions:

1. Ensure that the DDS has DC power cable installed
   See *page 44*
   The Icon lights are Off **{1}**
2. With Delta Off, place instrument in cradle.
   Take care that Analyzer Icon comes On (Green) **{2}**
   The main battery in the handle is charging although there is no indicator.
3. If Delta On, the Battery Status Screen appears
4. Place a spare battery in the auxiliary socket
   Battery Icon is On (Red - unless fully charged) **{3}**
5. Both batteries will charge; their real-time charge status is displayed.
6. When both batteries are fully charged, the Icons display Green. **{4}**

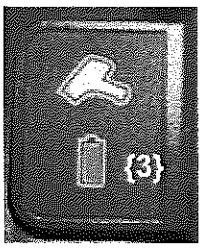


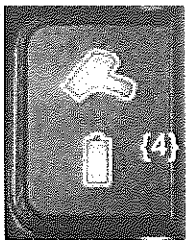


Delta Docking Station Procedure for
Automatic Cal_Check

1. Ensure that the DDS has DC power cable installed
   See *page 44*
   The all Icon lights are Off **{1}**
2. With Delta On, place instrument in cradle.
   Take care that Analyzer Icon comes On (Green) **{2}**
   Battery Status screen is displayed.
3. Ensure that the Battery Status screen stays On.

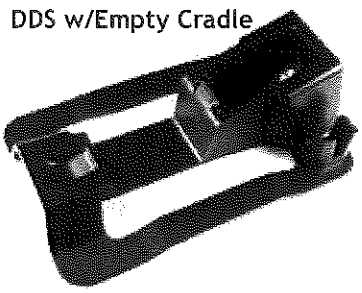
NOTE: Do not exit this screen -->> the automatic Cal_Check function will not work.



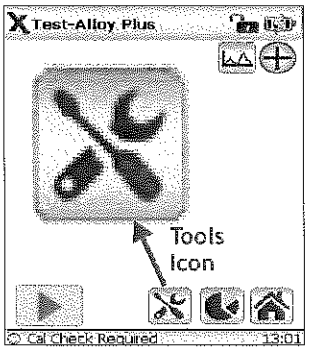


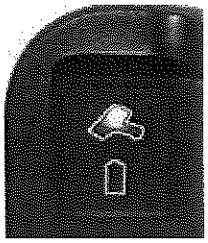
Use the Delta Docking Station for Startup - Initial Cal Check

DDS w/Empty Cradle



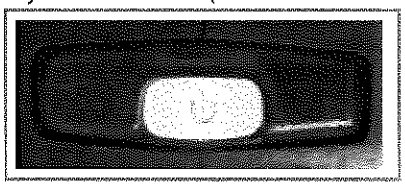
DDS w/Premium Delta in Cradle



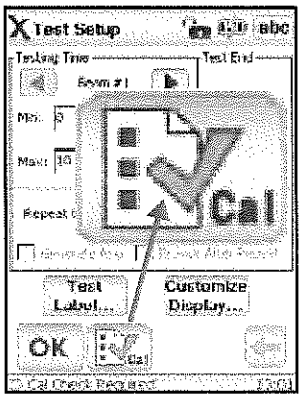


DDS - Indicator Light
Delta is engaged in cradle

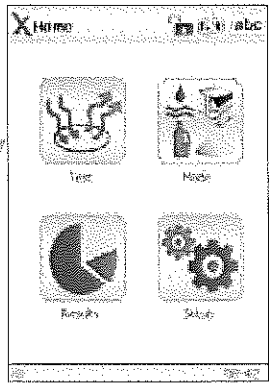
1. Place analyzer into the Delta Docking Station cradle.
   Ensure that the Delta indicator light is ON.
   This signifies that the instrument is properly seated in the cradle.
2. Press analyzer's I/O switch (>1 second to turn it ON.)



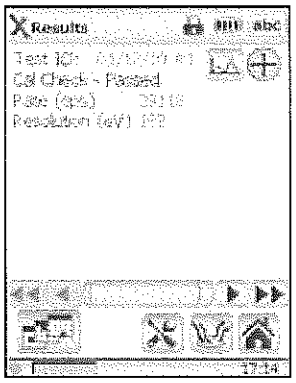
Radiation Safety **NOTICE** appears after a few seconds.



3. Read and respond to notice
   Tap **START** to acknowledge that you are a certified user.
   The UI begins its launch with the following messages:
   Initializing System
   Starting System
   Loading Files



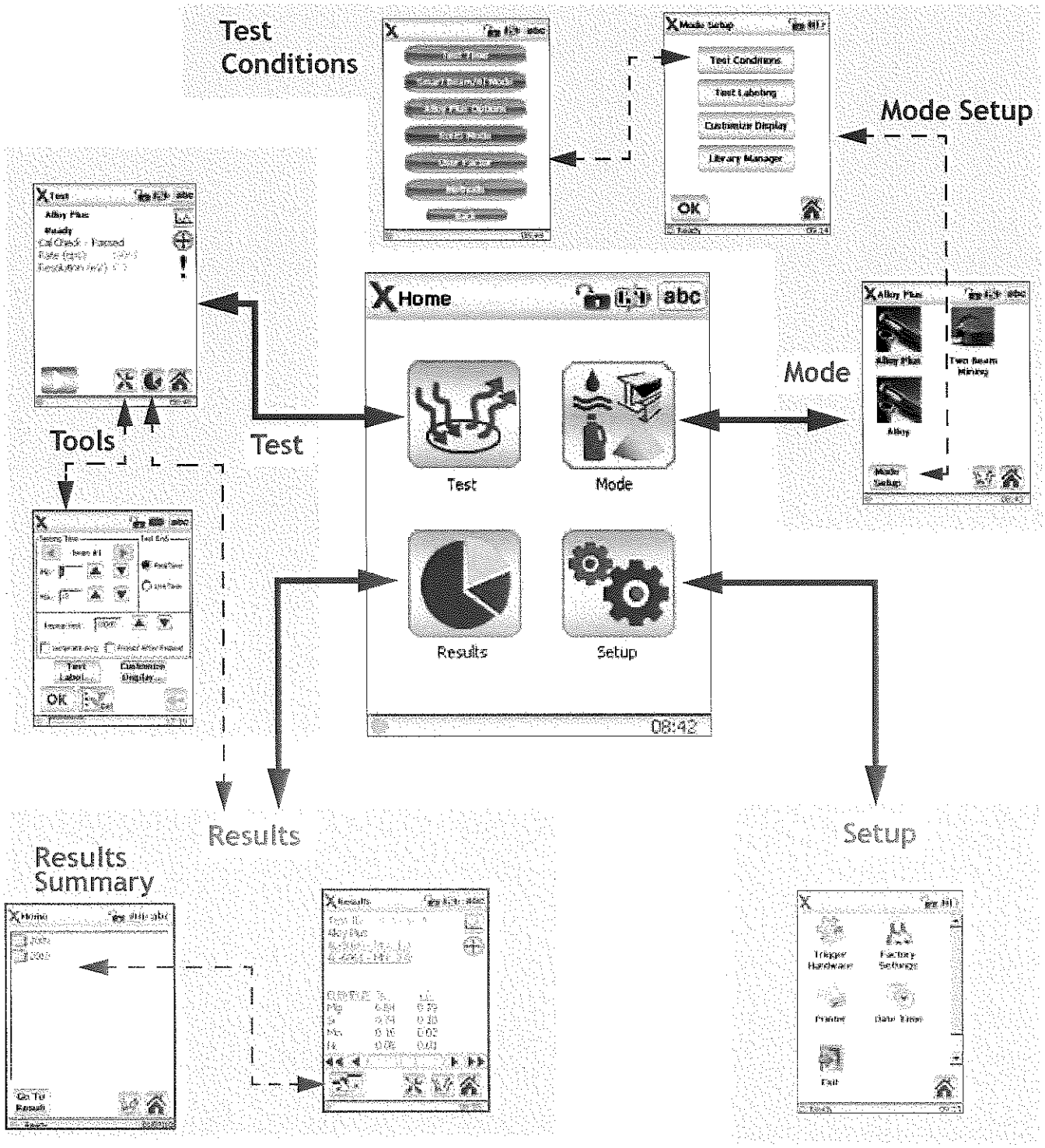
4. Unit displays a **Test** screen using the Mode last selected.
   4a. If the mode should be changed, go to **Home** screen.
   4b. Select the Mode button
   4c. Choose the desired Mode
5. Return to **Test** (now with your selected Mode)
   Note that message *Cal Check Required* is present.
6. Choose **Tools** icon:
   This launches **Test Setup** screen with Cal_Check button



7. Tap **Cal_Check**
   If necessary, unlock the **Trigger** with the icon at top of screen.
   Procedure begins immediately; it concludes in about 15 seconds.
8. Message *Cal Check - Passed* means you may begin testing.
   Message *Cal Check - Failed* will give error message, such as"! Wrong Count Rate"
   Re-try the procedure
   If unit fails repeatedly, contact InnovX service or your local distributor.

SNAPSHOT of Delta's User Interface

The Delta's user interface is introduced by the startup **Radiation Safety** and **Initialization** screens. Main operations then revolve around the **Home** screen.



Typical Test Procedure

Background

Details of routine testing operations vary depending on the selected analysis mode. Some relevant modes for Delta XRF users are:

• Alloy Modes —Alloy Plus —Fast ID & Pass/Fail —Precious Metals	• Mining Modes —Two-Beam Mining —Mining —Car Catalyst	• Soil Modes —Environmental —Exploration	• Consumer Goods —RoHS —Consumer Products

For the test sequence (below) the instrument has:

- Mode selected (Soil 3 Beam), and
- Cal Check procedure successfully completed.

To conduct a typical test:

1. Remove the instrument from the Delta Docking Station. Place the the measurement window flush against the sampling area. Ensure the sampling area is covered by the window.

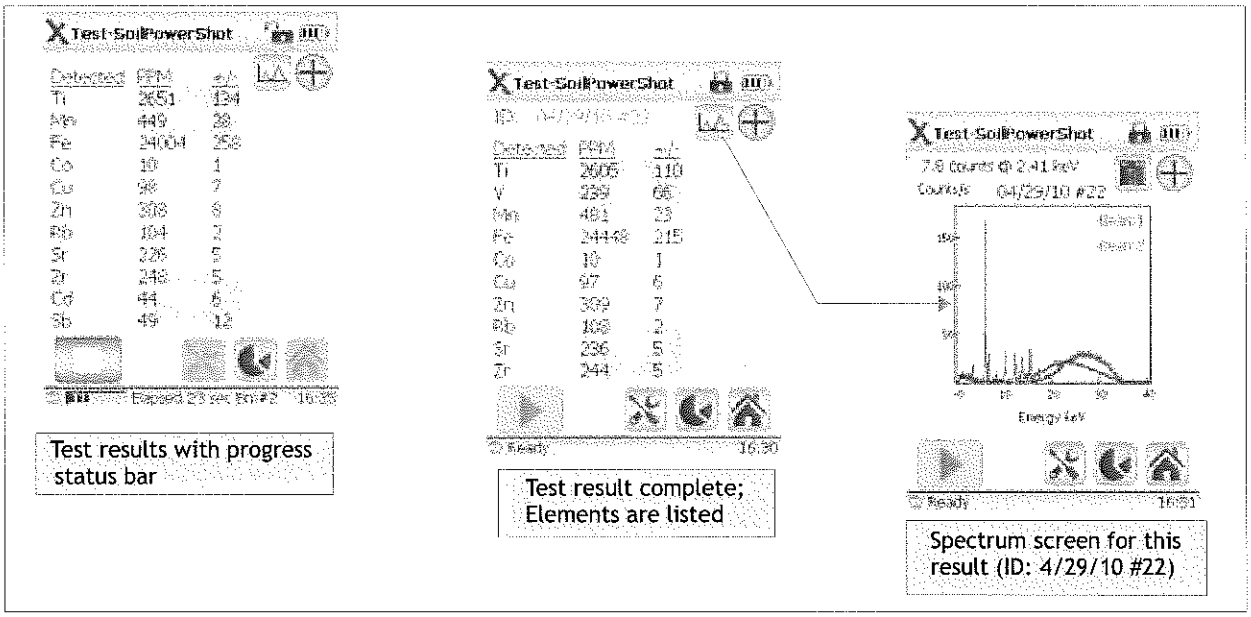
WARNING

Do not point the unit at yourself or any other person during operation. Do not test small samples in your hand. Place them on a surface for testing.
See *C2, Safety Information, Pages 29-31* for examples of safe and unsafe testing techniques.

2. Use one of these techniques to initiate the X-ray beam:
   a. Tap **Start Test (Green Arrow Icon)**, or
   b. Pull the trigger (toggles unit ON); can release the trigger during the test, or
   c. Pull-and-hold the trigger (deadman trigger function is enabled)
   This is a mandatory technique in Canada.

   Trigger options are configurable from:
   Setup > Trigger Hardware > Trigger Settings

The Test screens are as follow:



End of Day Operations

Save Results

When finishing testing for the day (or shift, or current session} InnovX recommends that test results be saved (e.g. exported) to a PC.

A necessary prerequisite is a DATA connection between the analyzer and PC.

This connection is made in one of two ways:

- Analyzer in DDS cradle — use the powered hub USB cable assembly (PN 103209 and PN 103210) from the DDS' rear Data port to a USB port of the PC
- Analyzer NOT in DDS cradle — use the mini USB B to USB A (PN 101310) cable from the analyzer's Data port to a USB port on the PC

The UI operational sequence is:

1. Navigate from **Home** > **Results** > **Calendar**
2. Select Year, Month, Day listing; it lists the total number of tests for the day
3. Select **Tools**, then **Results Setup**
4. Select the **Export** icon (button)
5. Choose the results to be exported
6. Name the export file (or accept default name)
7. Select Destination to save to
8. Tap the **Export** button

The file is exported.

GO TO

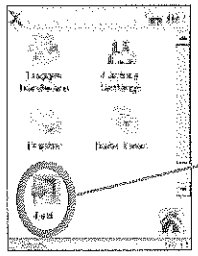
- See *"Delta SW User Interface Guide"* for the details (including options) of this sequence.

Ending Test Operations

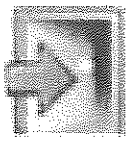
When testing and exporting are complete, the user has the following options:

- Turn off Delta with I/O switch or UI (see Exit Options procedure below); store unit in a secure location
- Place Delta in Docking Station and use the "Automatic" option:
  - Leave Delta powered ON;
  - Ensure that DDS is On (Delta ICON is lit);
  - Unit initiates Cal_Check after being idle for five minutes, then every ten hours thereafter.

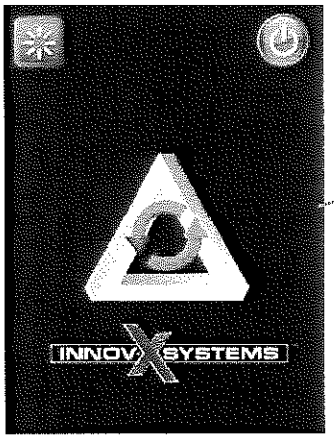
Exit Option Procedure from UI



Setup Screen



Exit Icon



User has **three** options:

Soft Reboot
Restart OS and restart IX app.

Power OFF
Turn analyzer off after confirmation.

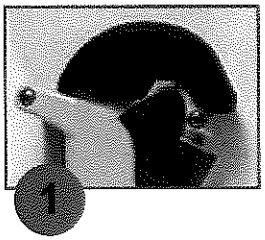
Relaunch
Restart IX app.

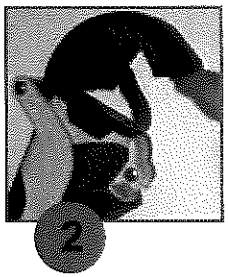
Battery Issues

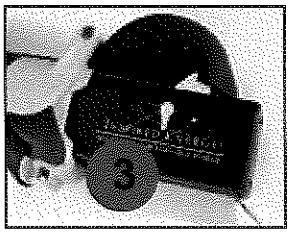
1 — Changing a Battery

To CHANGE the battery:

1. Hold the instrument by the handle, upside down, so the bottom of the instrument base is pointing upward with the nose pointing away from the operator.
2. Pull the rubber latch and lift cover.
3. Remove the existing battery using the tab. A new instrument will **not** have an installed battery.
4. Insert the charged battery into the analyzer with the battery connectors facing to the left. The battery slot is keyed so that the battery can be inserted only one way.







2 — Battery Status

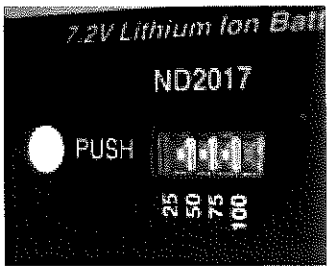
To TEST a Li-ion battery's charge status:

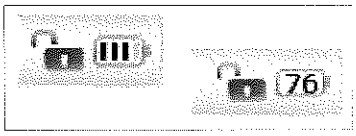
EXTERNAL battery test —

1. Push the white button on the battery.
   The green lamps indicate the percent of charge, from less than 25% to 100%.
2. If a battery has a charge of less than 25%, use the Delta Docking Station to establish a full charge. See *Page 45*.

INTERNAL MAIN battery test —

1. The battery icon from any UI screen (upper right side) shows an approximate value of charge.
2. Tap the battery icon and a more precise charge percentage is displayed as a number.



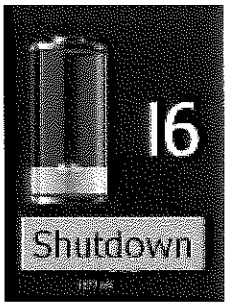


3 — HOT SWAP for Delta Battery

A battery HOT SWAP capability is a **standard feature** with the Delta analyzer. An operator can remove and replace a battery without having to shut down, restart, or Cal Check.

When the battery is removed:

- A "Shutdown" status display gives the percentage of internal charge remaining.
- If the internal charge reaches 0, you have to re-start the unit with the I/O switch, after inserting a fresh battery.
- If red X-ray indicator lights flash, the battery voltage is too low.



Cal_Check Information

Cal_Check Facts

Question: What is involved with the Cal_Check procedure?

Answer: The analyzer:

- Collects a spectrum on a known standard (Alloy 316 Stainless Steel)
- Compares a variety of parameters to values stored when the instrument was calibrated at the factory.
- When comparisons are within pre-set tolerances, the unit determines that it remains properly calibrated.

GENERAL FACTS INCLUDE:

- Cal_Check must be performed when the analyzer requests the procedure.
- The *Start* button and trigger are disabled until a successful Cal_Check is achieved.
- You can run a Cal_Check at any time during *InnovX* software operation, except during a test.
- When Cal_Check is in progress, the x-ray indicator light assembly blinks.This indicates that the X-ray tube is energized and the filter wheel is operational. In addition, a status bar appears on the UI display, showing the percentage completion for the measurement.
- The Cal_Check procedure takes about 15 seconds.

Cal_Check Procedures

There are two separate techniques:

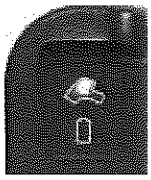
- **In the test laboratory** - Use the DDS to initiate "on-demand" procedure. Described above in *"Use the DDS for Startup - Initial Cal Check"*:

  Also have the "Automatic" option, as follows:

  Leave Delta powered On and InnovX software running;

  Ensure that DDS is On (Delta ICON is lit);

  Unit initiates Cal_Check after being idle for five minutes, then every 10 hours thereafter.



- **In the field** - Use the Coupon {the procedure is described below)

1. Place the 316 stainless steel Cal_Check coupon on a flat surface.

   See *C2. Safety Information, Page 29* for safety measures that must be observed.
2. Position the analyzer's measurement window flush over the coupon.
3. With the Test Setup screen invoked, tap the Cal Check icon. There are now two techniques to choose from:
   - Tap the "Start Test" icon, or
   - Pull the trigger (or pull-and-hold if using "Deadman Trigger")
4. The procedure takes about fifteen seconds.

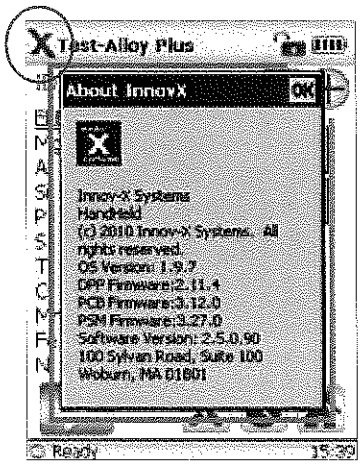
NOTE

Improperly positioning the window over the coupon can result in a failure.

5. When Cal_Check completes successfully, you may begin testing.
6. If Cal_Check fails, ensure that:
   - Coupon is positioned correctly.
   - X-ray indicator assembly is blinking during the procedure.
   - You have waited several seconds before starting the procedure.
7. If Cal_Check fails again,
   - Shut down the Delta software.
   - Shutdown and restart the analyzer.
   - Launch another attempt to Cal Check.

If Cal_Check fails repeatedly, contact Innov-X Systems Customer Service or your local distributor. Contact information is available at appendix *A6. Packing and Shipping*

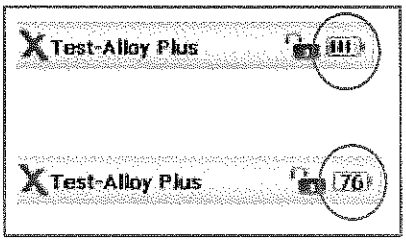
TIPS - or - things you should know about the Delta



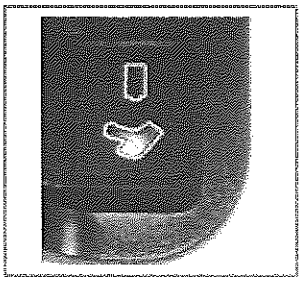
System Information
To call information "About InnovX" tap the X (InnovX icon) in upper left of UI screen. This provides various firmware and software versions that are installed on the analyzer.

UI Screen Note
All User Interface screens have a time-out (power saving) feature that causes the screen to go blank after 45 seconds if the UI is not accessed or the unit is not moved.
However, the analyzer is still running.
Restore the screen by tapping it or by moving the instrument.

Battery Status Info #1
When you turn on the instrument and you may not be aware that the battery is low, the X-ray indicator (Red LEDs) flashes dimly and briefly. The unit will not turn ON. Swap out the battery.



Battery Status Info #2
The on-screen battery icon (in upper right corner of UI) shows "real-time" battery charge status in a **graphical** way.
Tap this icon to receive a **numeric** value for battery charge level.



Delta Docking Station to Delta Analyzer: Contact Status
Keep the rubber boot attached to instrument when inserting it into the DDS. This helps ensure that the DDS contact pins are engaged.

The DDS analyzer icon (rear left corner) should be ON.

If the rubber boot is not available, and the analyzer icon remains OFF, use a small piece of padding under the handle to ensure contact.

C5. Alloy Analysis Modes

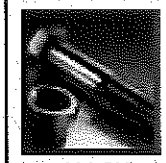
Alloy analysis for the Delta family includes:

- Wide range of modes and calibrations
- Outstanding performance for a variety of materials

There are five Alloy modes/calibrations:

ALLOY

Use with: **ALL Models**



ALLOY

Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, W, Hf, Ta, Re, Pb, Bi, Zr, Nb, Mo, Ag, Sn, Sb

Fundamental Parameters Analysis for metal alloys.
Alloy library including 300+ grade specifications, common tramp limits, including full editing capabilities.

ALLOY Plus

Use with: **Premier** and **Standard Only**

ALLOY PLUS

Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, W, Hf, Ta, Re, Pb, Bi, Zr, Nb, Mo, Ag, Sn, Sb
PLUS **Mg, Al, Si, P**

Fundamental Parameters Analysis for metal alloys.
Optimized beam condition for extended light element performance.
Alloy library including 300+ grade specifications, common tramp limits, including full editing capabilities.

FastID

Use with: **All** Models

Spectral signature matching for alloy grade & chemistry calculation.
Full library editing & alloy matching capabilities included.

Pass/Fail

Use with: **All** Models

Spectral signature or **chemistry matching** for alloy grades.
Customer created library with min/max grade specifications.
Full library editing & alloy matching capabilities included.

Precious Metals Additions

Use with: **All** Models

ADDS **Ir, Pt, Au, Rh,** and **Pd** to Analytical Analysis calibration suite.

Introduction to Alloy Analysis Modes

ALLOY

ALLOY PLUS

The Delta family of instruments currently presents six unique modes for alloy analysis. The core analytical analyzer modes/types are:

Alloy mode - **Classic** (PiN detector based) type

Alloy Plus mode - **Standard** (SDD detector based) type

- **Premium** (SDD detector based) type

All three analyzer types can support additional alloy-oriented modes, such as

FastID

Pass/Fail

Precious Metals (No added details)

Alloy analysis utilizes a Fundamental Parameters (FP) algorithm to determine elemental chemistry. This method calculates chemistry from the spectral data, without the requirement of stored fingerprints. The Analytical FP calibration is done at the factory, and requires no user set-up or recalibration. The software also searches an alloy grade library to produce a grade match based on the calculated chemistry. Analytical mode can provide a **grade ID** and **chemistry** in as little as one second, with increased precision for longer test times.

Alloy mode/Classic type supports Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, W, Hf, Ta, Re, Pb, Bi, Zr, Nb, Mo, Ag, Sn, Sb.

Alloy Plus mode /Standard type and Premium type supports light elements Mg, Al, Si, and P in addition to the core list of elements from the Alloy mode.

Standard and Premium units expand the Limits of Detection range permitting operators to analyze these light elements without a vacuum or helium purge requirement.

Both modes have a feature, Altitude Compensation, which automatically corrects calibrations based on barometric pressure.

Determination of Grade Identification:

Analytical modes utilize a Factory Grade Library consisting of a set of minimum and maximum values for each element in an alloy.

There is a SPECIFIC Alloy Factory Grade Library for EACH Delta model.

See appendix *A8. Alloy Grade Libraries* for a listing of the alloys that are contained in each Factory Grade Library.

Additionally, every analyzer is shipped with a "Tramp" library comprised of seven base alloys. These seven items with their min/max element values are increasingly valuable to fast and accurate sorting in Pass/Fail and FastID modes.

The libraries can be searched individually or together. All libraries, including each Factory Grade Library, can be edited by the user. However, InnovX strongly suggests that users NOT edit the Factory Grade Library. Instead, copy the Factory Grade Library to a USER library, then make any edits on it.

Match Number Concept

After calculating chemistry with the Fundamental Parameters algorithm, *Innov-X* compares the chemical composition values to grade tables stored in a grade library. The application calculates the value for a parameter called *Match Number*. This provides an indication of how close the measured alloy's chemistry is to library values.

- The *lower* the Match Number, the *better* the match.
- A Match Number of 0 is an *exact match*, meaning that the calculated chemistry for all elements falls within the grade table specifications.

GO TO

- See *"Delta User Interface Guide"* (PN 103202) for a complete description of the *Innov-X* application's User Interface.
- See *A8. Alloy Grade Libraries* for listing of each model's "Factory Grade Library"
  - A "Spectral Fingerprint" library that would be used for "FastID" and "Pass/Fail" analysis is created by the customer.

Match Issues

There are three Match determination possibilities provided within the Analytical modes:

EXACT MATCH

An unknown alloy is matched to one of the grades contained in the Grade Libraries, and a *Grade ID* appears on the *Results* screen. Often other grades are listed with their accompanying Match Numbers. The analyst has the opportunity to view their elemental chemistries and see how they differ from an exact match.

MULTIPLE MATCHES

In some cases, several grades are shown as possible matches. This can signify one of two conditions:

- There was not enough statistical information to definitively separate two or more alloys. The actual identification of the unknown alloy is one of the grades listed. Often increasing the testing time makes it possible to separate the alloys.
- There was sufficient statistical information, but the test sample did not meet any of the existing specifications with enough precision to cause an exact match identification.

NO MATCH

If no matches are found within the libraries, the words *NO MATCH* appear.

There are several causes for a NO MATCH result:

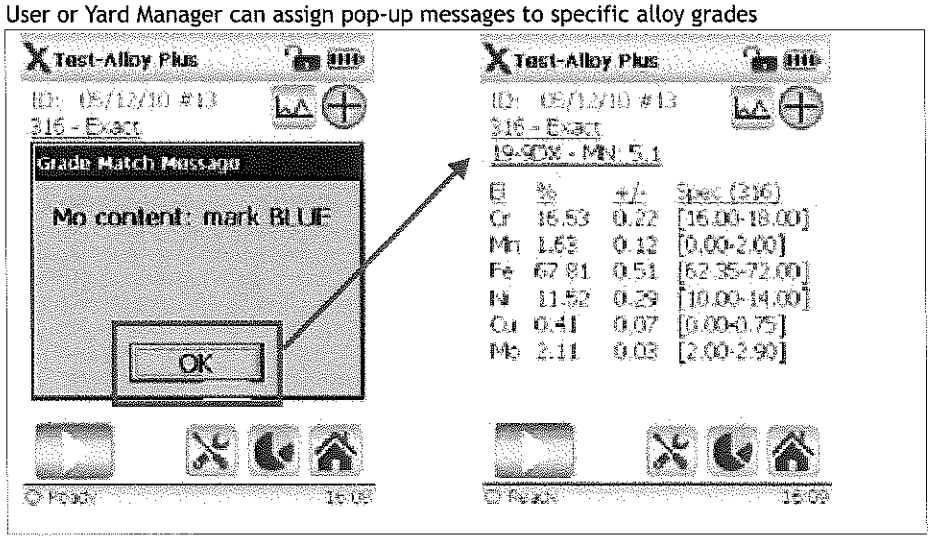
- The test sample does not meet any of the specifications in the Grade Library.
- The test sample is coated; Remove the coating by grinding, filing, or sanding and repeat the test.
- The testing time was too short.
  - Increase the testing time and measure the sample again.
- The Match Number is too low.
  - If possible, increase the Match Number

Scrap and Recycling Features

Delta analyzers in Alloy or Alloy Plus modes support many new features to specifically enhance scrap processing by maximizing speed and accuracy.

Grade Match Messaging (GMM)

User or Yard Manager can assign pop-up messages to specific alloy grades



- Quick start for next test, or
  view the chemistry details with one click

Grade Match Messaging offers:

- Immediate sorting instructions
- Less operator training
- More efficiency and higher throughput

SmartSort

Automated sorting decisions that allow users to maximize speed and sorting accuracy. Some features include:

- Specific grades set up to automatically extend testing time.
- Maximize efficiency for speed testing by automatically extending test time for light elements (Mg, Al, Si, P, S)
  - Eliminate unnecessary long tests

Nominal Chemistry

Nominal Chemistry looks for 'invisible' elements, based on grade ID, including:

- Elements not tested under active beam, (like Al in Beam 1)

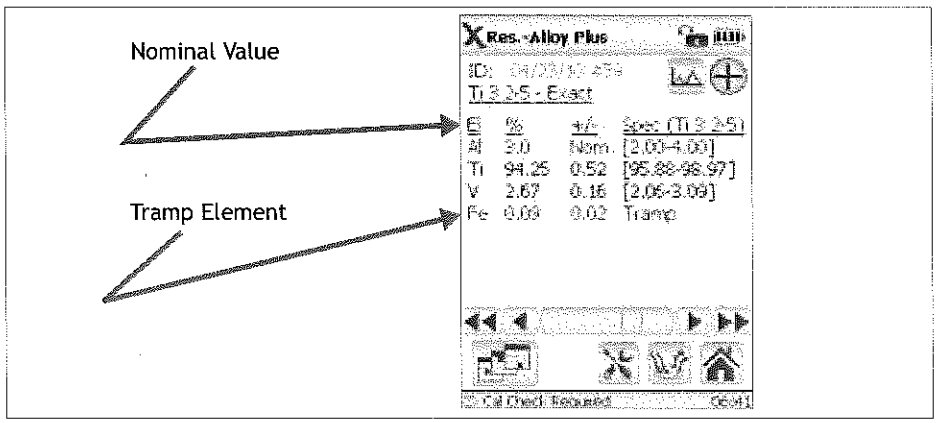
  — or —

- XRF invisible elements (like B or C)

Tramp Library

Analyzers with Alloy or Alloy Plus mode come pre-loaded with a tramp library based on industry standards.

- Operators may assign other "Tramp" elements with max tolerated concentration for individual elements in seven unique graded families.
- Analyzers can report tramp material (optionally) and simplify grade match by not counting small, expected amounts of tramp elements against the grade match.



See appendix *A8. Alloy Grade Libraries* for a discussion of the Tramp Library concept, including:

- How the InnovX Tramp Library works
- Practical advantages of the Tramp element approach
- List of seven Tramp Base Alloys

Test Sample Considerations

Coated or Painted Samples

XRF is a surface analysis technique, where X-rays penetrate a very short distance into most alloy samples. Therefore, the analyzer detects what is on the surface of an alloy, rather than what comprises the bulk of the material. If a material has been coated, plated, painted, or has had some sort of surface treatment, such as heat treating, it may be misidentified.

For example, a steel piece painted grey may show high concentrations of titanium from the paint, and may be misidentified as a titanium alloy. In another example, large amounts of metal dust or turnings on a surface may be detected by the analyzer.

To ensure proper identification of coated materials, grind an area slightly larger than the analyzing window to remove the coating. It is important to select the correct grinding material so as to not interfere with the analysis.

Do not use Silica for a Silicon analysis.

It may not be necessary to completely clean and polish all materials, however, remove obvious metal dust.

Mixed Samples, Heterogeneous Materials

Often finished metal pieces may consist of more than one type of metal. In addition, you may wish to measure mixed turnings, or an assortment of small pieces. In these cases, remember that the analyzer measures the entire area covered by the analyzing window and reports an average chemistry. For turnings, this is useful, as the analyzer provides an average composition. However, if two or more pieces of metal cover the window, the results is just an average reading, and may tell very little about the composition of one piece or the other.

When shooting metal pieces, or welds, ensure that **only** the metal of interest is covering the analyzing window.

Small and Irregularly Shaped Samples

To measure samples smaller than the analyzing window:

- Increase the testing time.

— and —

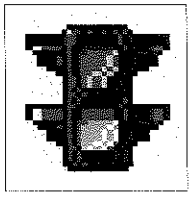
- Maximize the material in contact with the window.

The precision of analysis for small parts measurements is reduced, as the signal from smaller samples is less than for samples that completely cover the window. If possible, analyze the largest, flattest side of an irregularly shaped object.

GO TO

- See *A4. Typical Test Procedure* for description of a Test sequence.
- See *"Delta User Interface Guide"* (PN 103202) for a complete description of the *Innov-X* application's User Interface.

Introduction to FastID Mode (All Models)



FastID mode is designed to quickly identify an alloy. It uses an empirical calibration method known as a "type" calibration. It is most useful where the number of alloys to test is small and well known.

FastID is best suited for Positive Material Identification (PMI) and QA/QC applications.

For example, where alloy producers or fabricators handle materials that may be very similar or where maximum user simplicity is a primary concern.

This mode offers:

- Simplified results (grade name only or less information on tramp elements).
- A narrow, customized grade library base upon stored spectral fingerprint reference standards.
- **Full chemical analysis based on reference standard assays.**
- Results which are the best combination of SPEED and ACCURACY.
  - A Grade and Chemistry result in as little as one to two seconds.
- User selectable match criteria settings.
- Expansion of up to 500 additional alloy grades and assays (alloy chemistries) – password protected.
- Multiple independent grade libraries. You choose to search one or more libraries.
- All libraries are editable

How FastID works:

Prerequisite: The operator creates a "custom FastID fingerprint' library by testing an array of reference standards. This list spans the number of alloys for which he is interested.

1. Delta's XRF process allows a test sample to create a spectral fingerprint.
2. Analyzer compares this spectral fingerprint to entries from library of many certified spectra, the "custom FastID fingerprint" library.
3. Analyzer finds the best spectral match to the sample spectra: thus identifying and reporting the matching alloy grade.
4. If concentration data has been entered for the standards, the instrument then calculates the sample's **chemistry.**

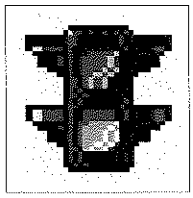
   The reported chemistry data are an extrapolation from standard intensity data stored in the customer -generated fingerprint library.

   The user gets a real time grade match and a precise report of the chemical composition of the sample.

NOTE

– Because FastID mode performs a spectral match to a library of reference standards, it is important that before testing, a "likely" stored reference standard be in the customer-created FastID fingerprint library.

Introduction to Pass/Fail Mode (All Models)



Pass/Fail mode is designed for high-throughput alloy sorting and quality control.

Mode Features

- All test samples are sorted by comparison to an operator-selected reference standard.
- Results are displayed as a *PASS* or a *FAIL*, depending on whether they match the reference standard.
- Pass/Fail criteria may be based on:
  - "quality of fit" to the selected spectral fingerprint

    — or —
  - elemental chemistry.
- Pass/Fail ranges may be implemented for one or more elements.
- This mode offers a full range of options from the simple sorting of mixed loads in a recycling facility to QC on specific element(s) of the most complex superalloys.

Pass/Fail mode has two options: ***Fingerprint** and **Chemistry***:

1. Fingerprint Option

Select this method when the goal is to determine whether or not test samples are a **specific grade**.

Fingerprint Pass/Fail and FastID use the same method to determine a match.

Data from analyzed samples are compared to the reference standard fingerprint.

If the differences between the fingerprints are small enough, the sample is judged to be of the same grade as the reference sample.

This method requires:

Only that the library contains a valid fingerprint for the reference standard.

2. Chemistry Option

Select this method to determine whether the chemistries for specific elements fall **within specified min/max grade** specifications.

Chemistry pass/fail process is:

1. Analyzer uses the fingerprint method to determine whether the sample matches the reference sample.
   If it does not, it automatically fails.
2. If Step 1 has a match, the alloy chemistry is calculated from assays stored for the standard fingerprint.
3. The calculated chemistry for each element is compared to the values stored in a *Grade Table*.

   In order for a sample to pass, all the chemistries must be within "n" standard deviations of the min and max values specified in the grade table.
   Number "n" is specified by the user.

This method requires three items:

(1) a valid fingerprint, (2) assays for that fingerprint, and (3) Min/Max values saved in the library.

C6. Mining Modes

There are three Mining modes

MINING Use with: **ALL models**

Mining

Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, W, As, Pb, Bi, Zr, Mo, Ag, Cd, Sn, Sb
(elements may be customized on request)
Fundamental Parameter based calibration for **ore grading and percent-level** analysis of **process bulk** samples.
Suitable for measurement of percentage level analyte concentrations, 0.5% and greater.

2 BEAM MINING Use with: **Premier** and **Standard** Only

Mining

Mg, Al, Si, P, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, W, As, Pb, Bi, Zr, Mo, Ag, Cd, Sn, Sb
(elements may be customized on request)
Mining mode optimized for **SDD based systems to enhance SPEED and LOD for light elements**

CAR Catalyst Use with: **All** Models

Mode ICON
T.B.A.

Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, W, Hf, Ta, Re, Pb, Bi, Zr, Nb, Mo, Ag, Sn, Sb
PLUS **Rh, Pt, Pd**
Accurate analysis of **bulk recycled catalyst** materials

The Mining modes/types are:

Mining mode - **Classic** (PiN detector based) type
2 Beam Mining mode - **Standard** (SDD detector based) type
Beam Mining mode - **Premium** (SDD detector based) type
Car Catalyst mode - **All** types

These modes utilize a Fundamental Parameters algorithm which automatically corrects for inter-element results.
The units can analyze:

- *in situ* (directly on the ground),
- prepared soil samples (in sample cups)
- bagged samples

Best Practices

Check Standards

Measure a check standard after each Cal Check, and periodically throughout the day. This confirms that data continues to be as accurate as possible.
The standards provided with Delta instruments are contained in special XRF sample cups. These cups have film windows (through which the soil can be viewed and analyzed) on one side, and solid caps on the other side.

Sample Presentation

in situ testing

In situ testing is performed by pointing the analyzer at the ground. Clear any grass or large rocks away and hold the analyzer with the probe head front flush to the ground. Since dirt can accumulate on the analyzer window, wipe the window clean after each analysis. Ensure the window is not ripped or punctured.

Bagged or prepared sample testing

Analyze prepared samples in a sample cup, through its Mylar window. Place the instrument's measurement window directly over the sample cup with the Mylar side up.
Preparation considerations include:

- Avoid measuring very thin samples, as this can affect results. Prepare samples cups to contain at least 15 mm of packed samples.
- When analyzing bagged samples, ensure that sufficient sample material exists in the bag to completely cover the window with a sample thickness of a minimum of 15 mm.
- When using bags, cheaper bags (having thinner plastic walls) are better than more expensive ones (which have thicker plastic walls).

Optional Accessories

Accessories that can assist in Mining mode testing are:

- A-035: Soil Foot
- 990055: Soil Extension Pole
- A-020-D: Workstation - portable, fully shielded, closed beam test stand for bench-top or remote controlled testing.
- Trimble Xplorer Package

Typical Test Procedure

GO TO

See *"A4. Typical Test Procedure"* for description of a Delta Test sequence..

Mining Mode Options

Test length in Mining Mode is **user defined.**

GO TO

Refer to *"Delta User Interface Guide, User Factors"* for procedure to modify User Factors..

Factors

Mining modes allow you to create your own set of **factors**, focusing on particular elements of interest or correcting for matrix effects.

You can make several different *Factor* tables, allowing analysis of a variety of samples.

Setting Mining User Factors

Example:

A group of samples covering the full concentration range for each element of interest are identified. Each sample is homogenized and split. A portion of each sample is sent to an outside lab for analysis. The other portion is analyzed with the analyzer. For best results ensure that the samples are very well homogenized, and characterized, so the correlation is quite good.

Procedure:

Plot the data. Innov-X data must be on the X-axis, lab values on the y-axis.

CAUTION

ENSURE that you use this order: Innov-X data on X-axis and lab data on the Y-axis.

Determine the linear best fit with both the slope and intercept for each element.

The slope and intercepts for these graphs are entered directly into the analyzer. In many cases it is sufficient to enter just a correction for the slope as the intercept is almost zero. In others, enter the slope and intercept.

You can enter multiple sets of user factors for different applications, or different ore bodies. A group of factors is given a name, and then the factors are entered. The factor set can then be selected by name.

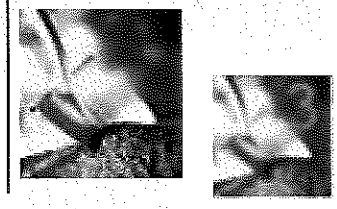
NOTES

C7. Soil Modes

The Delta family currently supports two soil modes, **Soil** and **3 Beam Soil**. Each mode has a possibility of two default element suites, **Environmental** and **Exploration.**
Note that both calibration packages cannot reside on one instrument.:

SOIL Environmental

Use with: **All** Models

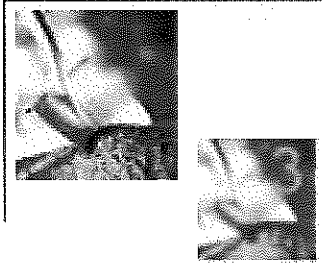


Soil 3 Beam

K, Ca, S, P, Cl, Ti, Cr, Mn, Fe, Ni, Cu, Zn, Hg, As, Pb, Se, Rb, Sr, Zr, Mo, Ag, Cd, Sn, Sb, Ba
(elements may be customize on request)
Compton Normalization algorithm designed for achieving lowest Limit of Detection (LOD) possible for **SOIL** and **BULK** samples.
PowerShot and SmartShot 3-beam modes included.

SOIL Exploration

Use with: **All** Models



Soil 3 Beam

K, Ca, S, P, Cl, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, W, Hg, As, Pb, Bi, Se, Th, Rb, U, Sr, Zr, Mo, Ag, Cd, Sn, Sb

(elements may be customized on request)
Compton Normalization algorithm designed for achieving lowest Limit of Detection (LOD) possible for exploration samples. PowerShot and SmartShot 3-beam modes included.

Soil Mode Beam Selection

SmartShot Beam Mode:

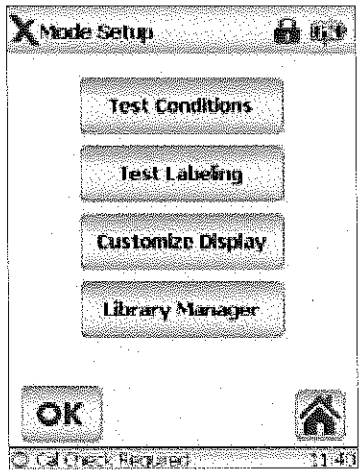
Uses a single incident bean setting (Transition Metals) otimized to deliver ultra fast results with solid LOD performance across the periodic table. SmartShot offers excellent sensitivity in the fastest testing time possible.

PowerShot Beam Mode:

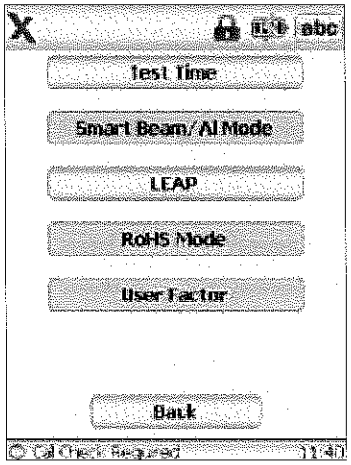
Offers a fully optimized, multi-beam analysis method that provides exceptional LODs for all elements analyzed - heavy metals, transition metals, and light elements. PowerShot can be used to analyze the full element range, or to focus in on a particular element of interest, such as Cr, Cd, Ni, or Cu. Any or all of the following beams conditions may be selected:

- Heavy Elements
- Transition Metals
- LEAP (Light Elements)

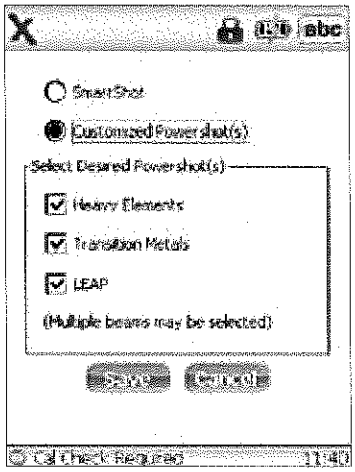
Use this procedure to configure beam selections:



From the Mode Setup screen select **Test Conditions.**



Select the **LEAP** option



Configure one or more **Powershot(s).** Note that multiple beams may be chosen.
Press **Save** to return to soil testing.

Best Practices

Check Standards

Measure a check standard after each standardization, and periodically throughout the day, for a minimum of one minute. Elemental concentrations for elements of interest, in the range expected at the site, plus or minus the error on the reading, should be within 20 percent of the standard value. *A2.Soil Testing* describes recommended quality assurance considerations in detail.

The standards provided with the analyzer are contained in XRF sample cups. These containers have a film window (through which the soil can be viewed) on one side, and a solid cap on the other side. Always measure samples through the film window.

Sample Preparation

Preparation considerations include:

- Avoid measuring very thin samples, as this can affect results. Prepare samples cups to contain at least 0.5"(usually 4-8 grams) of packed samples.
- When analyzing bagged samples, ensure that sufficient sample material exists in the bag to create a a sample thickness of a minimum of 15 mm for a spot size that is larger than the analyzer's measurement window.
- When using bags, cheaper bags (having thinner plastic walls) are better than more expensive ones (which have thicker plastic walls).

C8. Consumer Goods Analysis Modes

There are two consumer goods modes:

RoHS

RoHS

RoHS regulated elements- **Cr, Hg, As, Pb, Br, Cd,**
PLUS Cl, Ti, Fe, Co, Ni, Cu, Zn, Sn, Sb, Ba

Analysis software for measurement of restricted elements in electronics and consumer goods. Auto-compensations built in for metal, polymer, and mixed matrices.

Consumer Products

Consumer

Analysis software designed for CPSIA & Prop 65 testing. **Pb** content displayed as **Pass/Fail** based on regulated limits.

Additional elements Cl, Ti, Cr, Fe, Co, Ni, Cu, Zn, Hg, As, Br, Cd, Sn, Sb, Ba also reported.

Introduction to RoHS Mode

Toxic metals in consumer electronics are the focus of EU regulations that have worldwide ramifications. These new directives currently include:

- *Restriction of Hazardous Substances* (**RoHS**)
  - Designates maximum allowable levels of Pb, Cd, Cr^{6+}, Hg and certain Br-containing flame retardants (PBB and PBDE) in new electrical and electronic equipment sold into the EU.

The limits for RoHS elements are:

- <0. 1% Pb, Cr6+, Hg, Br (as flame retardants, PBB and PBDE)
- <0.01% Cd

The Innov-X analyzer is a screening tool for RoHS Compliance. It is used to:

- Directly analyze the amount of toxic metals in electronics,
- Identify quickly whether a plastic is made of or contains:
  - PVC
  - A brominated flame retardant.

XRF measures *total elemental composition*, regardless of speciation of the element. Therefore, it reports

- Total chromium including the concentration of hexavalent chromium plus any other forms of Cr.
- Total bromine, however cannot distinguish the type of brominated flame retardant present in analyzed materials.

In order for XRF to be quantitative, samples must be:

- Homogeneous
- Have a certain minimum sample thickness
  - Five (5) mm for polymers and light alloys
  - Fifteen (15) mm for liquid samples
  - One (1) mm for other alloys

If samples are heterogeneous, too thin, or too small, only qualitative screening is possible.

The IEC-ACEA (International Electro-technical Commission - Advisory Committee on Environmental Aspects) recommends XRF screening.

Test Overview

The Delta analyzer controlled by *InnovXPC* application software (in RoHS mode) automatically executes a test sequence to determine:

- Whether a sample is an **alloy**, **polymer**, or **mixed**.
  - "Mixed" indicates heterogeneous samples consisting of both polymer and alloy, such as wires or circuit boards.
- Whether each RoHS element **passes**, **fails**, or is **inconclusive** when compared to a set of stored criteria.
  - These criteria are either those recommended by the IEC, or ones added by the user.

The sequence begins with the instrument utilizing tube settings appropriate for analyzing a polymer sample. The following logic applies:

- If the sample is determined to be a polymer or mixed, the test continues, and a calibration based on a polymer matrix is used.
- If the sample is found to be a metal alloy, the analyzer switches to a secondary test, using an alloy matrix calibration, in order to determine correct alloy concentrations.

Check Standards

Innov-X Systems recommends that a check standard be measured after each Cal Check procedure, and periodically throughout the day.
Two certified standards are provided for verification.

- At least one standard should be measured for a minimum of two minutes.
- Concentrations for target elements (plus or minus the error on the reading) should be within 20% of the standard value.
- Standards provided are contained in XRF sample cups with a Mylar window (through which the plastic pellets can be viewed) on one side, and a solid cap on the other side.
- Samples should be measured in the sample cup, through the Mylar window.

Sample Presentation

Since many pieces of plastic analyzed for ROHS compliance are very small, take care to measure them in a safe and accurate manner. See the IEC-ACEA recommendations for minimum thickness of test samples.

IEC Quantitative Screening Requirements

RoHS requirements are derived from the *"Directive 2002/95/EC of the European Parliament and of the Council of the European Union on the restriction of the use of certain hazardous substances in electrical and electronic equipment."*
Dated 27 January 2003.

Important Current Issues

- At this User Manual's release date (May, 2010), the IEC requirements (including limits and exemptions) have **not been formally accepted.**
  A timetable for acceptance has not been established.
- Users must be aware that the information in Figure 1 concerning RoHS screening limits has been extracted from **proposed/draft** IEC-ACEA documentation.
- **Innov-X strongly advises users to have their own compliance departments determine the current status of the requirements that they must meet.**

Elemental Range/Limits for RoHS Compliance

—RoHS— Elements	PASS	Lower Limit	Incon-clusive	Upper Limit	FAIL
		Polymer Materials			
Cd	P	≤(70-3s)	< X <	(130 +3s)≤	F
Pb	P	≤(700-3s)	< X <	(1300+3s)≤	F
Hg	P	≤(700-3s)	< X <	(1300+3s)≤	F
Br	P	≤(300-3s)<	X		
Cr	P	≤(700-3s)<	X		
		Metallic Materials			
Cd	P	≤(70-3s)	< X <	(130 +3s)≤	F
Pb	P	≤(700-3s)	< X <	(1300+3s)≤	F
Hg	P	≤(700-3s)	< X <	(1300+3s)≤	F
Br			N/A		
Cr	P	≤(700-3s)<	X		
		Electronics			
Cd	P	LOD	< X <	(150 +3s)≤	F
Pb	P	≤(500-3s)	< X <	(1500+3s)≤	F
Hg	P	≤(500-3s)	< X <	(1500+3s)≤	F
Br	P	≤(250-3s)<	X		
Cr	P	≤(500-3s)<	X		

Figure 1: Proposed Screening Limits for RoHS Elements

Grade Definitions for Screening

Grade	Proposed Screening Criteria
PASS	Results for ALL elements are lower than the lower limits shown in Figure 1.
FAIL	Result for ANY element higher than the higher limits shown in Figure 1.
INCONCLUSIVE	Result of the quantitative analysis, for any of the elements Hg, Pb, or Cd, is in the region defined as intermediate, OR if the result of the elements BR and Cr is higher than the higher limits shown in Figure 1, the analysis is inconclusive. Additional investigation must be performed.

GO TO

- See *A4. Typical Test Procedure* for description of a Test sequence.
- See *"Delta User Interface Guide"* (PN 103202) for a complete description of the *Innov-X* application's User Interface.

Introduction to Consumer Products Mode

Background

This mode is dedicated to testing items for Lead (**Pb**) content.

The result output is **Pass/Fail** based on the regulated limits that are specified in:

- CPSIA (the Consumer Products Safety Improvement Act of 2008)
- Prop 65 - refers to California Proposition 65 enacted in 1986

Additional elements that may be reported are:
Cl, Ti, Cr, Fe, Co, Ni, Cu, Zn, Hg, As, Br, Cd, Sn, Sb, Ba

Regulatory limits vary with the governing body, state-to-state, and country-to-country. European Union nations generally use the RoHS limits and testing practices.

The regulatory limits for Pb are:

- CSPIA: 300ppm currently, but going to 100ppm
- RoHS: see Table 1.0 on Page 70

GO TO

- See *A4. Typical Test Procedure* for description of a Test sequence.
- See *"Delta User Interface Guide"* (PN 103202) for a complete description of the *Innov-X* application's User Interface.

A1. Overview: X-Ray Fluorescence (XRF) Spectrometry

Basic Theory

Although most commonly known for diagnostic applications in the medical field, x-rays are the basis of many powerful analytical measurement techniques, including X-ray Fluorescence (XRF) Spectrometry.

XRF Spectrometry determines the elemental composition of a material. This method identifies elements in a substance and quantifies the amount present of those elements. An element is defined by its characteristic X-ray emission wavelength (λ) or energy (E). The amount of an element present is determined by measuring the intensity of its characteristic line.

All atoms have a fixed number of electrons (negatively charged particles) arranged in orbitals around their nucleus. The number of electrons in a given atom is equal to the number of protons (positively charged particles) in the nucleus. In the classical Periodic Table of Elements, the Atomic Number is specified by the number of protons. Each Atomic Number is assigned an elemental name, such as Iron (Fe), with Atomic Number 26.

XRF Spectrometry typically utilizes activity in the first three electron orbitals, the K, L, and M lines, where K is closest to the nucleus. Each electron orbital corresponds to a specific and different energy level for a given element.

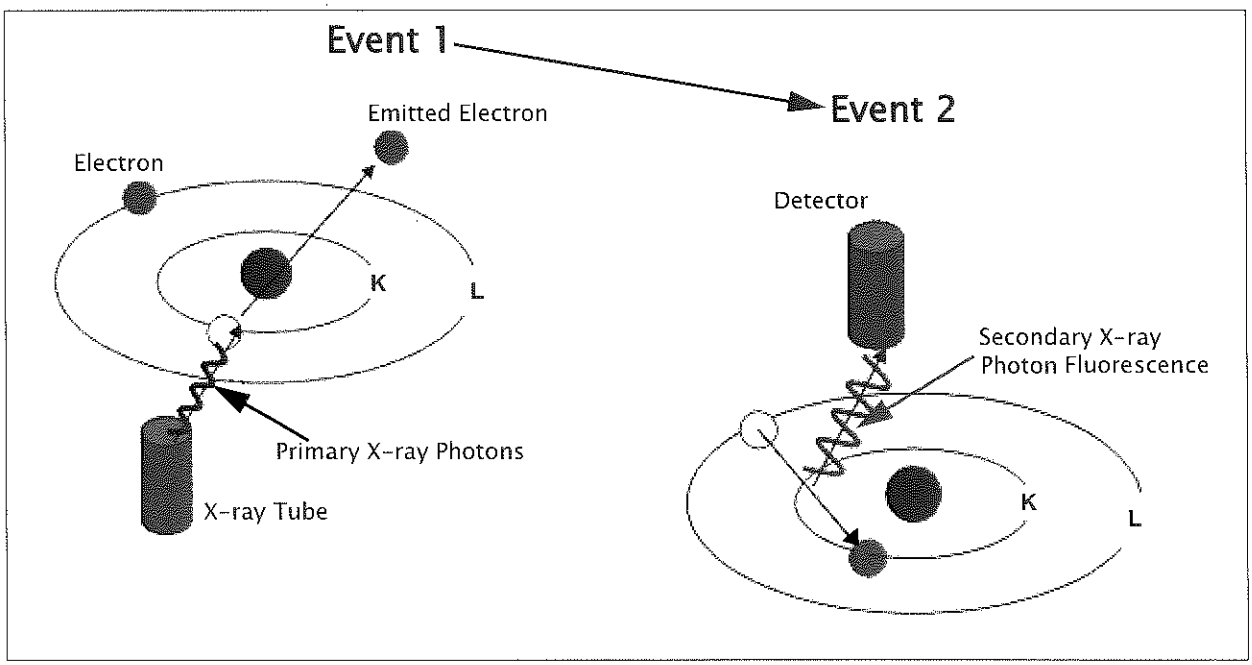
In XRF Spectrometry, high-energy primary X-ray photons are emitted from a source (X-ray tube or *radioisotope*) and strike the sample. The primary photons from the X-ray source have enough energy to knock electrons out of the innermost, K or L, orbitals. When this occurs, the atoms become unstable ions. Electrons seek stability; therefore, an electron from an outer orbital, L or M, moves into the newly vacant space at the inner orbital. As the electron from the outer orbital moves into the inner orbital space, it emits an energy known as a secondary X-ray photon.

This phenomenon is called fluorescence.

The secondary X-ray produced is characteristic of a specific element.

The energy (**E**) of the emitted fluorescent X-ray photon is determined by the difference in energies between the initial and final orbitals of the individual transitions.

This is described by the formula $E=hc/\lambda$ where **h** is Planck's constant; **c** is the velocity of light; and λ is the characteristic wavelength of the photon.

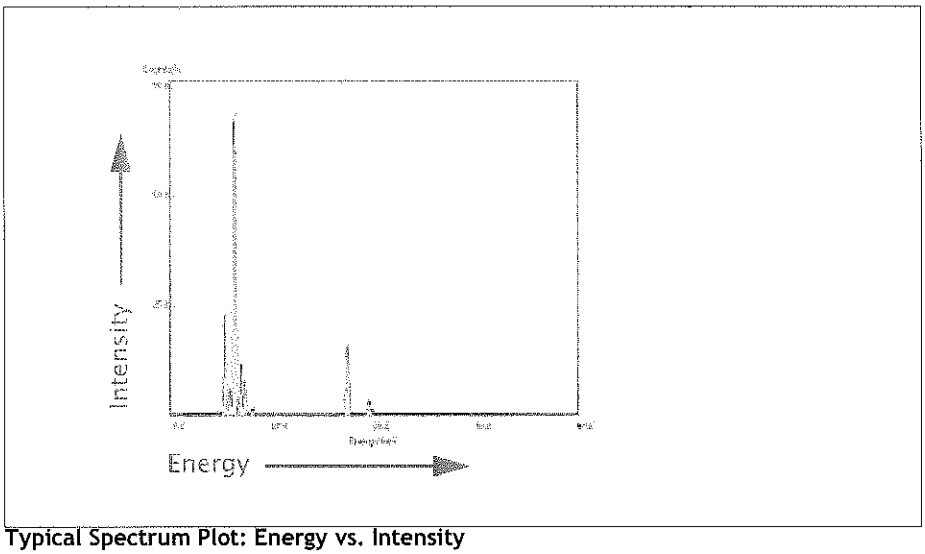


Creating a Secondary X-ray - Photon Fluorescence

Wavelengths are inversely proportional to the energies; they are characteristic for each element.

For example, the Ka energy for Iron (Fe) is about 6.4keV. The number of element-specific characteristic X-rays produced in a sample over a given period of time, or the intensity, is measured. This determines the quantity of a given element in that sample.

Typical spectra for EDXRF Spectrometry appear as a plot of **Energy (E)** versus the **Intensity (I)**.



Typical Spectrum Plot: Energy vs. Intensity

History

Timeline for XRF Spectrometry

- Wilhelm Roentgen discovered X-rays in 1895.
- Henry Moseley first published methods for identifying and quantifying elements using XRF in 1913.
- XRF research and development continued, especially during WWII.
  - Critical developments in the aircraft, automotive, steel, and other metals industries increased the need to identify alloys *quickly* and *reliably*.
- The first commercial XRF Spectrometers became available in the early 1950's. These systems were based on **Wavelength Dispersive (WD) XRF** technology.
  - The characteristic **wavelength** of an element was measured **one element at a time.**
  - WDXRF systems were useful for elemental analyses, however, the equipment had the following properties:
    - Large size
    - High initial cost
    - Required highly skilled operators to use and maintain them.
- In the late 1960's, **Energy Dispersive (ED) XRF** technology emerged as a viable commercial choice:
  - EDXRF measured the characteristic **energy** of an element.
  - Improvements in solid state detectors offered better energy resolution of the signal.
  - Had potential to collect and display information on **all** of the elements in a sample at the **same time.**
- Many of the early EDXRF systems used radioisotopes for excitation. They had the following properties:
  - Required changing sources to determine all the elements of interest.
  - Did not easily resolve multiple elements in a single analytical run.
- The current state-of-the-art in EDXRF is the result of
  - Advancements in technology (particularly X-ray tubes, solid-state components, electronics, computers, software)
  - Application of the technology by instrument manufacturers, research scientists, engineers, and industrial users.
- Now a mature technology, XRF Spectrometry is routinely used for R&D, QC, production support, and regulatory compliance.

Elemental Analysis

Investigators involved with elemental analysis generally have two working instrument techniques – *Wet Chemistry* and *XRF Spectrometry*. They are compared operationally as follows.

Wet Chemistry

Important considerations are:

- Instrument techniques are *time-consuming*.
  - Often takes twenty minutes to several hours for specimen preparation and analysis.
- Specimen is *destroyed*.
- Often necessary to employ *concentrated acids* or other *hazardous materials*.
- Requires *disposal* of waste streams generated during the analytical process.
- Relatively *high cost* per sample.

However, wet chemistry instrument techniques are necessary if the primary measurement requirement involves elemental concentrations in the PPB (or lower) range

XRF Spectrometry

Important considerations are:

- Easily and quickly identifies and quantifies elements over a wide dynamic concentration range, from PPM levels up to virtually 100% by weight.
- Does *not destroy* the sample.
- Overall sample turnaround time is *fast*.
  - Requires little, if any, specimen preparation.
  - Often results are available within seconds, minutes for some details.
- Relatively *low cost* per sample

Interferences

All elemental analysis techniques experience chemical and physical interferences. They must be corrected or compensated for in order to achieve adequate analytical results.

WET CHEMISTRY ISSUES

Most suffer from interferences that are corrected only by extensive and complex specimen preparation techniques.

XRF SPECTROMETRY ISSUES

The primary interference is from other specific elements in a substance that can influence (matrix effects) the analysis of the target element(s) of interest.

However, this interference style is well known and documented.

Both types of analyzer techniques benefit from (a) instrumentation advancements, and (b) mathematical corrections in the system's software.

In certain cases, the *geometry* of the sample can effect XRF analysis.

- This is compensated for by:
  - Grinding or polishing the sample
  - Pressing a pellet
  - Making glass beads

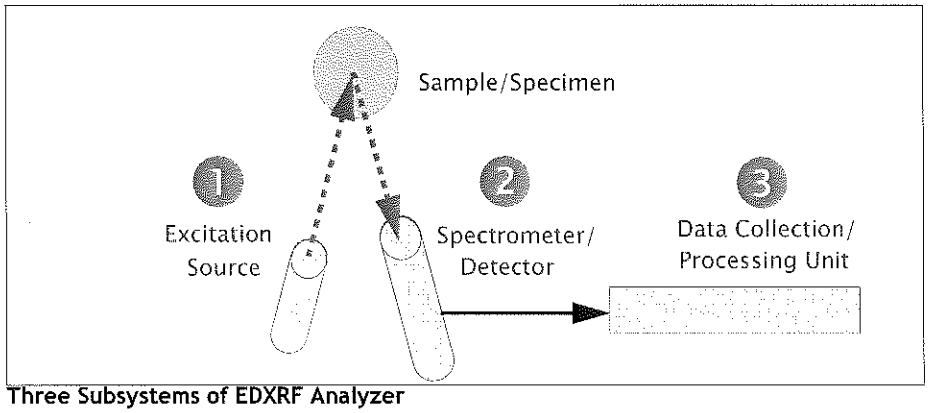
Quantitative analysis

XRF Spectrometry supporting quantitative analysis typically employs one of two software applications:

- **Empirical Methods**
  Uses calibration curves derived from standards similar in property to the target unknown sample.
- **Fundamental Parameters (FP)**
  FP is frequently preferred because it allows elemental analysis to be performed *without* standards or calibration curves.
  The analyst can use the system immediately.
  Modern computers support this *no-standard* mathematical analysis, FP, accompanied by stored libraries of known materials. These systems quickly determine not only the elemental composition of an unknown material, but even identify the unknown material itself.

EDXRF Spectrometers

An EDXRF instrument typically has three major subsystems:



Three Subsystems of EDXRF Analyzer

EDXRF analyzers are mechanically very simple; there are *no moving parts* in the excitation and detection subsystems. However, a bench-top analyzer can have moving parts.

When compared to WDXRF systems, EDXRF systems exhibit the following attributes:

- Ease of use
- Rapid analysis time
- Lower initial purchase price
- Substantially lower long-term maintenance costs

EDXRF analysis equipment is useful for many applications, including:

- Environmental analysis
- RoHS/WEEE compliance
- Scrap alloy sorting
- Forensic science
- Archaeometry

NOTES

A2. Soil Testing

This appendix explains usage of all of the company's hand-held portable analyzers with the ***Soil*** or ***3 Beam Soil*** mode option installed.

This document offers instructions/procedures and regulations, as well as useful reference material, regarding:

- Portable XRF equipment usage in accordance with accepted methods.
- Basic overview of the technique of x-ray fluorescence (XRF).
- Appropriate data quality assurance protocols.
- Sample preparation steps for operators analyzing prepared soil samples.
- Tables of certified values for selected standards.

If LEAP mode is enabled, refer to configuration help in *PN 103202*.

Section 1: Commonly Accepted Methods for Field Portable XRF

A commonly accepted method is shown: ***Field Portable XRF Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment.***

Features of this method are:

- It is a field screening method, for analysis of *in-situ* or bagged samples.
- The method provides basic quality assurance methods, including calibration verification, determination of instrument precision, accuracy and limit of detection.
- The method recognizes that some XRF instruments do not require site-specific calibrations by the operator, that is, the factory calibration provides appropriate data quality.
- The method recommends that a minimum of 5-10% of samples tested by XRF be confirmed by an outside laboratory, using a total-digestion EPA analytical reference method.

The purpose of this method is ***NOT*** to replace laboratory analysis.

There are two primary sources of error in assessing a site for metal concentration: *Analytical error* and *Sampling error.*

Analytical Error

The error in the analysis of any one sample by whatever technique is used, for example XRF, ICP, or AA.

Sampling Error

This arises when **too few** samples are collected and tested.

In this case an incomplete picture of the extent of metals contamination may be obtained. Although any one sample may be analyzed with very high analytical accuracy, measuring too few samples may result in contamination plumes being mis-judged in size, or depth into the soil. In extreme cases contamination can be missed entirely.

Methods have been developed to reduce Sampling Errors by **increasing** the number of samples measured. In general, a large number of screening-level measurements provide a better characterization of contamination than a small number of measurements produced by sample removal and analytical analysis. A large number of in-situ samples provide detailed data on contamination profiles, depth (provided surface soil is moved aside), and approximate contamination levels. Portable XRF can provide results with a high degree of analytical accuracy on any given sample.

Section 2: Overview of Field Usage:

Field portable XRF is generally used in three ways to test for metals in soil:

IN-SITU SOIL TESTING:

The XRF is placed directly onto the ground for soil testing. Operators remove any plant growth and foreign objects so that the analyzer probe is flush to the soil.

BAGGED SOIL SAMPLE TESTING:

A soil sample is collected in a thin plastic bag (i.e. a *Baggie*) and testing occurs directly through the Baggie. Except for a few elements - namely Cr, V and Ba - testing through the thin plastic bag has little effect on the test result. However, results for Cr, V and Ba will be lower by 20-30%.

PREPARED SOIL SAMPLE TESTING:

Prepared sample testing assures the operator of the maximum possible accuracy. Prepared sample tests require a sample to be collected, dried if necessary, sieved and ground into a powder. The prepared sample is then placed into a baggie or XRF cup for analysis.

Sample prep procedures are provided on Section 8: Sample Prep Procedures and Testing Protocols on page 130.

ALL analytical methods require a *uniform, homogenous* sample for the best results. **XRF is no different!**

The methods generally used, namely In-situ and bagged sample testing, are considered *field-screening methods*. Although a field-screening method, in-situ testing is a valuable technique because it generates a great deal of data very quickly. Prepared soil samples generally offer the best accuracy, albeit with several minutes of sample preparation required per sample.

Subsection 2-A: Data Quality Objectives

The objective of testing is generally to determine the mixture of in-situ versus prepared sample testing. It is important to understand your data quality objectives (DQO) in order to determine the appropriate mix of field screening and prepared sample testing.

In-situ testing usually provides only *screening-level data quality.*

This is because analytical testing always requires a uniform, homogeneous sample matrix. A laboratory achieves this by digesting the sample into a hot acid before analysis. Testing directly on the ground does not ensure that uniformity is met. Preparing a sample provides a uniform sample and likely better analytical data quality, although several minutes of testing time is required.

Most portable XRF operators use a mixture of in-situ and prepared sample testing. The exact mixture of in-situ and prepared sample testing depends upon the goals of the soil testing. The examples below serve as guidelines.

Example 1: **Initial site investigation to provide detailed contamination data with efficient use of laboratory analysis costs.**

PROBLEM:

The site needs to be assessed for metals contamination. Little information is available about what metals are present, likely contamination levels or geographic profile of contamination.

The goal of testing is to determine what metals are present at what levels, both in area and in depth into soil. Additionally, testing will locate possible contamination plumes and/or possible sources of contamination.

RECOMMENDED TESTING PLAN:

This example uses predominately in-situ testing. The analyst will perform in-situ testing, and gather samples into plastic bags for XRF analysis. A testing grid should be established in two or three dimensions, every several feet. XRF tests can be taken at each location or bagged samples can be collected from each location for later analysis. The in-situ data for each element analyzed may be plotted in a 2-dimensional grid (X, Y coordinates versus elemental concentration) to profile a site. These concentration profiles are ideal for showing contamination patterns, boundaries and plumes. Combining this data with historical use data from the site often allows the operator to deduce sources of contamination. Obtaining this level of geographic data with purely laboratory analysis would produce excessive analytical costs.

Prepared sample analysis should also be done to confirm the regions where in-situ data indicates low or non-detected levels of metal contaminant. There is little need to prepare areas where in-situ testing indicates high concentration levels.

Innov-X recommends this procedure:

For locations where in-situ tests indicate low or non-detected concentrations, calculate the total number of in-situ tests, collect 5% of this number of tests from the various locations. Prepare these samples according to instructions on "Section 8: Sample Prep Procedures and Testing Protocols on page 130." Use these prepared samples to confirm the findings of the in-situ testing. Send a subset of these prepared samples to a laboratory for confirmatory results.

COST JUSTIFICATION:

To adequately characterize a site may require 100-200 samples/acre to be sure the contaminated areas are firmly established. This work may be done with in-situ testing to generate laboratory savings of $5,000 - $10,000/acre depending upon the number of elements being analyzed. The cost reduction in off-site analysis often justifies the price of the XRF.

Example 2: **Monitor remediation efforts and assure site meets clearance levels before contractors leave the site.**

GOAL:

Minimize remediation costs by only treating contaminated soil, and obtain immediate verification that various site locations meet clearance objectives.

Recommended Testing Plan:

This type of project uses a lot of both in-situ and prepared sample testing. Use in-situ testing to thoroughly delineate contamination regions in both area and depth. To determine depth profiles, test surface soil, remove at least 1-2', and retest. Repeat this step as necessary to profile contamination depth to guide remediation activities (XRF is a surface technique and only analyzes the first few mm of soil sample). As part of clearance, collect several samples from *cleared* area. Prepare samples according to *"Section 8: Sample Prep Procedures and Testing Protocols"* on page 92. Test with portable XRF.

If XRF indicates that concentration levels are:

- In excess of clearance requirements, then continue remediation efforts.
- Below clearance requirements, then discontinue remediation efforts, and send a subset of the samples to an analytical laboratory to confirm results. Most operators safely assume that the cleanup requirements have been met for the elements in question, but await final analysis from the laboratory.

If XRF lists concentration levels as non-detected, but the detection level reported exceeds clearance requirements, send samples to a laboratory for final results.

Cost Justification: In-situ results are used to guide remediation efforts, in order to obtain maximum efficiency. Efficiency is produced because contamination boundaries are firmly established, thus avoiding remediation efforts with *clean* soil. Prepared sample testing is used to assure that clearance requirements are met on-site in near real-time (pending laboratory confirmation). Costs savings are generated by avoiding clearance failures. The contractors can leave the site earlier and will not be called back to the site for additional cleanup.

Important Note:

Never clear a site based solely on in-situ testing. Always use well-prepared samples to make a clearance decision.

Example 3: Minimize volume of hazardous waste for treatment or disposal.

Goal:

For some cleanup projects, the cost of soil disposal in a hazardous waste landfill is much greater than disposal in a standard landfill. Testing soil samples with XRF may minimize the amount of *clean* soil that is inadvertently shipped to a hazardous-waste landfill.

Recommended Testing Plan:

This example is almost entirely based on prepared sample testing. Representative samples are removed from the soil being hauled to landfill. Obtaining an accurate analysis of the samples is crucial for making a hazardous versus non-hazardous determination. For this reason, prepared sample testing is strongly recommended.

Important Note:

These types of samples are subject to Toxicity Characteristic Leaching Procedures (TCLP) for the landfill determination. In general, 20 times the XRF result should be less than the allowable limit for the metal in question. Please contact Innov-X Systems for more details on testing samples versus TCLP regulatory requirements.

Section 3: Quality Assurance

Quality assurance is detailed for both the proper use of the analyzer and for verifying the data quality of in-situ testing. All operators should perform the QC procedure, regardless of their data quality objectives. There must be strict requirements about quality assurance. Additionally, Innov-X recommends that operators verify the data quality of in-situ test results, if they are using in-situ data to guide their reporting or remediation decisions. Procedures are listed below:

Proper verification of instrument operation

Quality assurance here consists of testing known standards to verify calibration, as well as testing blank standards to determine limits of detection and to check for sample cross-contamination or instrument contamination. We recommend a detailed procedure, which is provided here in abbreviated form.

Components of instrument QC:

- An energy calibration check sample at least twice daily
- An instrument blank for every 20 environmental samples
- A method blank for every 20 *prepared* samples
- A calibration verification check sample for every 20 samples
- A precision sample at least one per day
- A confirmatory sample for every 10 environmental samples

Energy Calibration Check: The Innov-X analyzer performs this automatically; this is the purpose of the standardization check when the analyzer is started. The software does not allow the analyzer to be used if the standardization is not completed.

Instrument Blank: The operator should use the SiO_2 (silicon dioxide) blank provided with the analyzer. The purpose of this test is to verify there is no contamination on the analyzer window or other component that is *seen* by the x-rays. We recommend an instrument blank at least once per day, preferably every 20 samples. For either in-situ or prepared-sample testing, the operator should just test the SiO_2 blank to be sure there are no reported contaminant metals.

Method Blank: The purpose of the method blank is to verify that cross-contamination is not introduced into samples during sample preparation. We recommend following the sample preparation procedures with clean SiO_2 once very 20 prepared samples. This QC step is not required if the operator is not preparing samples.

Calibration Verification: Innov-X provides standard reference samples for calibration check by operator. The operator should perform a two minute test on a standard. The difference between the XRF result for an element and the value of the standard should be 20% or less. Calibration Verification should be performed upon instrument startup and periodically during testing.

NOTE

Innov-X recommends a calibration check every 4 hours. Some users make a calibration check every 20 samples. Reference standards are generally applicable for Pb, As, Cr, Cu, Zn. Innov-X provides additional reference standards for Priority Pollutant metals including Cd, Se, Ag, Hg, Ag, Ba, Sn, Sb, and Ni.

Precision Verification: It is good practice to make a minimum of one precision sample run per day by conducting from 7 to 10 replicate measurements of the sample. The precision is assessed by calculating a relative standard deviation (RSD) of the replicate measurements for the analyte. The RSD values should be less than 20 percent for most analytes, except chromium, for which the value should be less than 30 percent.

Confirmatory Sample: It is recommended that one confirmatory sample is run for every 10 samples collected. It is good practice to have confirmatory samples collected from the same sample material that is analyzed on site, but are sent to an off-site laboratory for formal analysis. The purpose of a confirmatory sample is to judge the accuracy of the data obtained by analysis on site and to allow corrections, if necessary."

Important Notes about confirmatory samples:

Innov-X always recommends that customers compare prepared-sample results to laboratory results. To do this, collect and prepare a sample following the protocols shown on *"Section 8: Sample Prep Procedures and Testing Protocols"* on page 92. Take a subsample and submit to the laboratory for analysis. The single largest error in XRF analysis is lack of sample preparation. For the best comparison, always use prepared samples.

Determining data quality of in-situ testing:

For operators relying extensively on in-situ testing, it is important to determine the data quality of this testing at a given site. *This protocol is not intended for every sample, but rather for a small percentage of samples considered representative of the site.* If the operator can demonstrate that quantitative data is achieved with little or no sample preparation, then the site characterization will be completed much more quickly but correctly.

For example, an operator may be able to demonstrate that the XRF result changes considerably when samples are passed through a 2 mm sieve, but that XRF results do NOT change appreciably upon finer sieving. In this case, the operator can conclude that good XRF data is achievable with only 2 mm sieving. Sieving only to this level requires far less time than a more robust sample preparation.

A protocol to determine the *appropriate level of sample preparation* is the following:

1. Delineate a region of soil approximately 4" x 4".
2. Perform several in-situ tests in this area, or collect the top (approximately) quarter inch of soil from this region, bag the soil, test through the bag. In either case, average the results.
3. If you did not bag the in-situ test sample, collect the top (approximately) quarter inch of soil from this region and sieve through the 2 mm sieve provided. Otherwise sieve the bagged sample used for the in-situ test. Thoroughly mix the sieved sample, and place some of the sieved material into an XRF cup, and perform a test of this sample.
4. If the results of this prepared sample differ by:
   - Less than 20% with the average in-situ result, this indicates the soil in this region is reasonably homogeneous. The data quality in this case is probably at the semi-quantitative level, rather than just screening data.
   - More than 20%, this indicates the soil is not very homogeneous, and there are serious particle size effects affecting your in-situ measurements.
     In this case, sieve the sample through the ~250 m sieve. Mix this sample and place a sub-sample into an XRF cup for testing. If this result differs from the previous by less than 20% then this indicates that at a minimum the 2 mm sieving is necessary to achieve higher data quality.
     If this result differs by more than 20% from the sample sieved through 2 mm, then particle size effects are still affecting the XRF result. In this case samples should be sieved through 125 μm to assure data quality at the quantitative level.

Section 4: Calibration for Innov-X Portable XRF

The Innov-X analyzer can run *three* different calibration methods, described below.

COMPTON NORMALIZATION:

In nearly all cases, customers use the Compton Normalization method. This method (recognized in EPA 6200) offers speed, ease of use, and generally good accuracy for concentration ranges from the ppm level up to 2-3% concentrations. As most field-testing is seeking to remediate or locate environmental contaminants, the upper limit of the calibration (2-3%) is generally not a limitation.

FUNDAMENTAL PARAMETERS (FP):

If customers do require a calibration up to 100% concentration (i.e. a pure element), then Innov-X recommends they also include the Fundamental Parameters (FP) software module with the analyzer. The FP module may be added at time of purchase or as an upgrade at any later date.

NOTE

In general, customers do not need to calibrate Innov-X analyzers for soil testing. The analyzer is delivered with a factory calibration, generally based upon the Compton Normalization (CN) method. The CN method has been proven over the past several years to provide a robust calibration generally independent of site-specific soil matrix chemistry.

All customers should follow the QC procedure described in Section 3, which includes a check of the calibration.

EMPIRICAL CALIBRATION:

In this case, customers run standards to generate calibration curves for various elements in specific soil matrices. With a well-prepared sample, the empirical method generally yields the most accurate result. In our experience, the accuracy gains going *from* Compton Normalization *to* Empirical Mode are small and not worth the extra effort in setting up calibration curves.

The empirical calibration module is an optional software package, available for an upgrade fee at the time of purchase, or as an upgrade at any later date.

CAUTION

The *greatest source of error* for in-field XRF analysis of soil is *lack of adequate sample preparation*, thus there is little gained in developing a sophisticated empirical calibration if the operator does not grind and homogenize the all measured samples.

Calibration Requirements:

The concentration of an element in a soil sample is well-described by the formula:

$$w_i = \frac{k_i}{M(Z,i)} I_i$$

where:

k_i = calibration constant for element *i*

ω_i = concentration of element *i* - the quantity being measured

I_i = measured x-ray intensity from element *i*

M(Z,I) = Soil matrix value

The factory calibration determines the value of the calibration constants k_i for each element, and a typical value M(Z,I). The calibration method - either CN, fundamental parameters, or empirical - performs the necessary corrections to the value M(Z,I) that are important for the site-specific soil chemistry. The XRF analyzer uses the measured intensity of each element's fluorescence from the sample, and the calibration data, to produce elemental concentrations.

Compton Normalization Calibration:

The Compton Normalization method calibration consists of the analysis of a single, well-characterized standard, such as an SRM or SSCS. The standard data are normalized to the Compton peak. The Compton peak is produced from incoherent backscattering of X-ray radiation from the excitation source and is present in the spectrum of every sample. The matrix affects the way in which source radiation is scattered off the samples. This scatter is directly related to the intensity of the Compton peak. For that reason, normalizing to the Compton peak can reduce problems with matrix effects that vary among samples. Compton normalization is similar to the use of internal standards in analysis for organic analytes.

Fundamental Parameters Calibration:

The fundamental parameters (FP) calibration is a *standardless* calibration. Rather than establishing a unit's calibration curve by measuring its response to standards that contain analytes of known concentrations, FP calibration relies on the known physics of the spectrometer's response to pure elements to set the calibration. Built-in mathematical algorithms are used to adjust the calibration for analysis of soil samples and to compensate for the effects of the soil matrix. The FP calibration is performed by the manufacturer, but the analyst can adjust the calibration curves (slope and y-intercept) on the bases of results of analyses of check samples, such as SRMs which are analyzed in the field.

Empirical Calibration:

The empirical calibration method requires that a number of site-specific calibration standards (SSCS) are used to establish calibration parameters. The instrument response to known analytes is measured and used to create calibration curves. Empirical calibration is effective because the samples used closely match the sample matrix. SSCSs are well-prepared samples collected from the site of interest in which the concentrations of analytes have been determined by inductively coupled plasma (ICP), atomic absorption (AA), or other methods.

The standards should contain all the analytes of interest and interfering analytes. Manufacturers recommend that 10 to 20 calibration samples be used to generate a calibration curve. The empirical method is the least desirable calibration method as it requires that new standards and curves are generated for each site that is analyzed.

Section 5: Effects of Moisture on XRF Results

Sample moisture has two effects on XRF results:

- It alters the soil chemistry, since water is another chemical compound that comprises the soil matrix.
- Moisture impedes the ability to properly prepare samples.

There is a further testing consideration:

- Laboratory results are provided on a *dry weight* basis.

EFFECT ON SOIL CHEMISTRY:

While the presence of significant moisture does impact the soil chemistry, modern XRF analyzers all perform automatic corrections for variations in soil chemistry from site to site. Indeed, such variations are expected, and that is the reason analyzers use Compton Normalization or fundamental parameters, in order to correct for moisture content changes as well as other differences in soil geochemistry.

It is known that moisture content above 20 percent may cause problems, since moisture alters the soil matrix for which the FPXRF has been calibrated. However, the Compton Normalization or fundamental parameters methods are implemented in order to automatically correct results for changes to the soil matrix. Thus, we believe that soil moisture is not a significant effect on accuracy due to effects of soil matrix, except for the *dilution* effect that can cause discrepancies with laboratory results which is described below.

SAMPLE PREPARATION ISSUES:

The inability to adequately prepare a wet sample is, we believe, the single biggest contributor to errors when testing wet samples. It is very difficult to grind or sieve a wet sample. The highest quality XRF results are generally obtained from prepared samples.

CAUTION

If the operator is unwilling to dry the sample to prepare it, comparisons to the laboratory may yield poorer correlation since the samples are not homogeneous.

Laboratory Tests on Dry-Weigh Basis:

Laboratories always dry samples prior to analysis. They report percent weight content based upon a dry sample basis. Portable XRF may often be used to analyze wet samples in the field, and results are thus reported that include the moisture content. Thus, with all other factors the same, the laboratory will report results higher than portable XRF. The results are higher by the amount of moisture content in the sample. For example, laboratory results will be 10% higher compared to XRF results, if the sample contained 10% by weight water when it was tested with XRF. Recall, this applies to samples where other possible sources of error are the same or negligible.

Section 6: Comparing XRF Results to Laboratory Results

Innov-X strongly recommends that operators compare prepared sample results to laboratory results. This is because prepared-sample results yield the best possible accuracy with portable XRF. Moreover, the most common source of error is due to non-uniform samples. No analytical technique, including the XRF technique, can properly account for non-uniform sample types.

To perform a comparison between XRF results and laboratory:

1. Collect a sample and prepare it according to the sample preparation guide given in *"Section 8: Sample Prep Procedures and Testing Protocols"* on *page 90*.
2. Take a sub-sample (5-10 grams) of the fully-prepared sample, place it into an XRF cup and perform at least a one-minute test on that sample.
3. Send the same sample to the laboratory for wet chemistry analysis.
4. Require the laboratory to use a *total-digestion method*. If the laboratory does not, they may not extract all of the elemental metal from the sample. In this case, the lab result will be lower than the XRF result. Incomplete sample digestion is one of the *most common* sources of laboratory error, thus it is very important to request a total digestion method.

Example of Error: The operator collects a bag of sample, performs XRF analysis on one part of the bag, and sends the bag, or part of the bag of sample to a laboratory for analysis. The laboratory reports a very different value than the operator obtained with the XRF.

Problem:

Since the sample is very non-homogeneous, the operator did not obtain a result that was representative of the entire bag of sample. The lab analyzed a different part of the sample and obtained a very different result due to the non-uniformity of the sample. The solution to this problem is, at a minimum, to test several locations in the bag of sample and report the average value. Also note the differences between the tests, as this is indicative of the non-uniformity of the sample. The operator should send entire bag of sample to the lab, and instruct the lab to prepare the sample before removing the sub-sample for lab analysis.

Best Practice:

The operator should homogenize and prepare the entire bag of sample, and then collect a sub-sample for XRF testing. After testing, the *same sample* should be sent to the lab.

Section 7: Common Interferences

Interference occurs when the spectral peak from one element overlaps either partially or completely with the spectral peak of another.

Case 1 Conditions
If the instrument is calibrated for both elements, one causing the interference and one being interfered with, it is generally capable of correctly handling the interference. In this instance, the element being interfered with may be measured with a poorer detection limit or poorer precision, but the analytical results should still be acceptable for field-portable XRF.

Case 2 Conditions
If the XRF is not calibrated for the element causing the interference, then the instrument may report the presence of elements not in the sample, or greatly elevated concentrations of elements in or not in the sample.

Example CASE 1: Lead and arsenic
Lead and arsenic. Most XRFs are calibrated for lead and arsenic. Lead interferes with arsenic (not vice-versa though). The net effect is a worsened detection limit for arsenic, and poorer precision. The XRF handles the correction automatically, but the precision is affected. The loss of precision is also reported by the XRF.
(Refer to Innov-X Applications Sheet: *In-field Analysis of Lead and Arsenic in Soil Using Portable XRF* which is now available as *Arsenic and Lead in Soil.PDF* for more detail).

Example CASE 2: Bromine
Bromine in the sample, but the XRF is not calibrated for bromine. Bromine, as a fire retardant, is being seen more and more in soil and other sample types. For this reason, Innov-X analyzers include Br in the calibration data. If Br is not calibrated, but is present in the sample, the analyzer will report highly elevated levels of Pb, Hg and As. The levels will depend upon the concentration of Br in the sample.
Interferences between elements can be broadly categorized into two types:

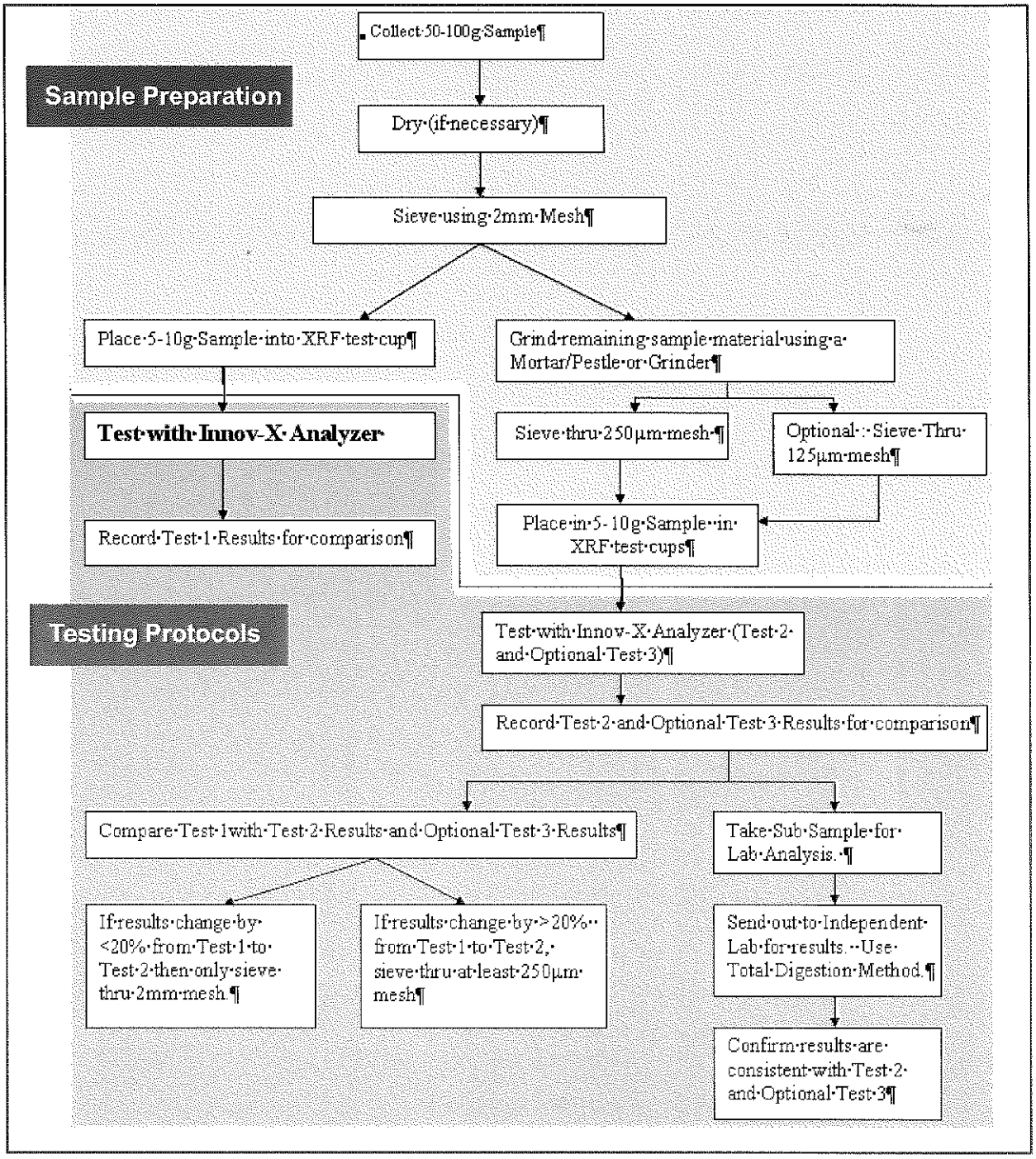
Z, Z-1, Z+1 interferences

Occurs when high levels of an element of atomic number Z are present. This can cause elevated levels of elements with atomic number Z-1 or Z+1. Generally, portable XRFs have good correction methods, so this interference only causes problems with very high levels of the element in question. Example: High concentrations of Fe (Z=26) in excess of 10% may cause elevated levels of Mn or Co (Z=25 or Z=27 respectively).

K/L interferences

Occurs when the L-shell line of one element overlaps with the K-shell spectral line of another element. The most common example is the lead/arsenic interference where the L-alpha line of lead is in nearly the exact same location as the K-alpha line of arsenic.

Section 8: Sample Prep Procedures and Testing Protocols



Section 9: NIST Certificates of Analysis

Innov-X systems provides three NIST standards (subject to change with availability). Each standard's certified values are given in Table 1 and Non-certified values are in Table 2 in the graphics below.

- ☑ SRM2709
- SRM2710
- SRM2711

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2709

San Joaquin Soil

Baseline Trace Element Concentrations

The Standard Reference Material (SRM) is intended primarily for use in the analysis of soils, sediments, or other materials of a similar matrix. SRM 2709 is an agricultural soil that was oven-dried, sieved, radiation sterilized, and blended to achieve a high degree of homogeneity. A unit of SRM 2709 consists of 50 g of the dried material.

The certified elements for SRM 2709 are given in Table 1. The values are based on measurements using one definitive method or two or more independent and reliable analytical methods. Noncertified values for a number of elements are given in Table 2 as additional information on the composition. The noncertified values should NOT be used for calibration or quality control. Analytical methods used for the characterization of this SRM are given in Table 1 along with analysts and cooperating laboratories. All values (except for carbon) are based on measurements using a sample weight of at least 250 mg. Carbon measurements are based on 100 mg samples.

Noncertified Values: Noncertified values, shown below, are provided for information only. An element concentration value may not be certified if a bias is suspected in one or more of the methods used for certification or if two independent methods are not available.

Table 2. Noncertified Values

Element	Mass Fraction (%)	Element	Mass Fraction (µg/g)
Carbon	[illegible]	Cerium	42
		Cesium	5.3
		Dysprosium	3.5
		Europium	0.9
		Gallium	14
		Gold	0.3
		Hafnium	3.7
		Holmium	0.50
		Iodine	5
		Lanthanum	23
		Molybdenum	2.0
		Neodymium	19
		Rubidium	96
		Samarium	3.6
		Scandium	12
		Thorium	11
		Tungsten	2
		Uranium	3
		Ytterbium	1.6
		Yttrium	18
		Zirconium	160

Table 1. Certified Values

Element	Mass Fraction (%)			Element	Mass Fraction (µg/g)		
Aluminum	7.50	±	0.06	Antimony	7.9	±	0.6
Calcium	1.89	±	0.05	Arsenic	17.7	±	0.8
Iron	3.50	±	0.11	Barium	968	±	40
Magnesium	1.51	±	0.05	Cadmium	0.38	±	0.01
Phosphorus	0.062	±	0.005	Chromium	130	±	4
Potassium	2.03	±	0.06	Cobalt	13.4	±	0.7
Silicon	29.66	±	0.23	Copper	34.6	±	0.7
Sodium	1.16	±	0.03	Lead	18.9	±	0.5
Sulfur	0.089	±	0.002	Manganese	538	±	17
Titanium	0.342	±	0.024	Mercury	1.40	±	0.08
				Nickel	88	±	5
				Selenium	1.57	±	0.08
				Silver	0.41	±	0.03
				Strontium	231	±	2
				Thallium	0.74	±	0.05
				Vanadium	112	±	5
				Zinc	106	±	3

SRM2709

☑ SRM2710

SRM2711

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2710

Montana Soil

Highly Elevated Trace Element Concentrations

This Standard Reference Material (SRM) is intended primarily for use in the analysis of soils, sediments, or other materials of a similar matrix. SRM 2710 is a highly contaminated soil that was oven-dried, sieved, radiation sterilized, and blended to achieve a high degree of homogeneity. A unit of SRM 2710 consists of 50 g of the dried material.

The certified elements for SRM 2710 are given in Table 1. The values are based on measurements using one definitive method or two or more independent and reliable analytical methods. Noncertified values for a number of elements are given in Table 2 as additional information on the composition. The noncertified values should **NOT** be used for calibration or quality control. Analytical methods used for the characterization of this SRM are given in Table 3 along with analysts and cooperating laboratories. All values (except for carbon) are based on measurements using a sample weight of at least 250 mg. Carbon measurements are based on 100 mg samples.

Noncertified Values: Noncertified values shown below are provided for information only. An element concentration value is not certified if a bias is suspected in one or more of the methods used for certification, or if two independent methods are not available.

Table 2. Noncertified Values

Element	Mass Fraction (%)
Carbon	3

Element	Mass Fraction (mg/kg)
Bromine	6
Cerium	57
Cesium	107
Chromium	39
Cobalt	10
Dysprosium	5.2
Europium	1
Gallium	14
Gold	0.6
Hafnium	3.2
Holmium	0.6
Iodine	5.1
Lanthanum	34
Molybdenum	10
Neodymium	22
Rubidium	120
Samarium	7.6
Scandium	8.7
Strontium	330
Thallium	1.3
Thorium	12
Tungsten	95
Uranium	25
Ytterbium	1.3
Yttrium	23

Table 1. Certified Values

Element	Mass Fraction (%)			Element	Mass Fraction (mg/kg)		
Aluminum	6.44	±	0.08	Antimony	38.4	±	3
Calcium	1.25	±	0.03	Arsenic	626	±	38
Iron	3.38	±	0.10	Barium	707	±	51
Magnesium	0.853	±	0.042	Cadmium	21.8	±	0.2
Manganese	1.01	±	0.04	Copper	2950	±	130
Phosphorus	0.106	±	0.015	Lead	5532	±	80
Potassium	2.11	±	0.11	Mercury	32.6	±	1.8
Silicon	28.97	±	0.18	Nickel	14.3	±	1.0
Sodium	1.14	±	0.06	Silver	35.3	±	1.5
Sulfur	0.240	±	0.006	Vanadium	76.6	±	2.3
Titanium	0.283	±	0.010	Zinc	6952	±	91

SRM2709

SRM2710

☑ SRM2711

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2711

Montana Soil

Moderately Elevated Trace Element Concentrations

This Standard Reference Material (SRM) is intended primarily for use in the analysis of soils, sediments, or other materials of a similar matrix. SRM 2711 is a moderately contaminated soil that was oven-dried, sieved, radiation sterilized, and blended to achieve a high degree of homogeneity. A unit of SRM 2711 consists of 50 g of the dried material.

The certified elements for SRM 2711 are given in Table 1. The values are based on measurements using one definitive method or two or more independent and reliable analytical methods. Noncertified values for a number of elements are given in Table 2 as additional information on the composition. The noncertified values should **NOT** be used for calibration or quality control. Analytical methods used for the characterization of this SRM are given in Table 3 along with analysts and cooperating laboratories. All values (except for carbon) are based on measurements using a sample weight of at least 250 mg. Carbon measurements are based on 100 mg samples.

Noncertified Values: Noncertified values, shown below, are provided for information only. An element concentration value may not be certified, if a bias is suspected in one or more of the methods used for certification, or if two independent methods are not available.

Table 2. Noncertified Values

Element	Mass Fraction (%)	Element	Mass Fraction (µg/g)
Carbon	2	Bromine	5
		Cerium	69
		Cesium	6.4
		Chromium	47
		Cobalt	10
		Dysprosium	5.6
		Europium	1.3
		Gallium	15
		Gold	0.3
		Hafnium	7.3
		Holmium	1
		Iodine	1.3
		Iridium	3
		Lanthanum	40
		Molybdenum	1.6
		Neodymium	31
		Rubidium	110
		Samarium	5.9
		Scandium	9
		Thorium	14
		Tungsten	3
		Uranium	3.6
		Ytterbium	2.7
		Yttrium	25
		Zirconium	230

Table 1. Certified Values

Element	Mass Fraction (%)			Element	Mass Fraction (µg/g)		
Aluminum	6.53	±	0.09	Antimony	19.4	±	1.8
Calcium	2.88	±	0.08	Arsenic	105	±	8
Iron	2.89	±	0.06	Barium	726	±	38
Magnesium	1.05	±	0.03	Cadmium	41.70	±	0.25
Phosphorus	0.086	±	0.007	Copper	114	±	2
Potassium	2.45	±	0.08	Lead	1162	±	31
Silicon	30.44	±	0.19	Manganese	638	±	28
Sodium	1.14	±	0.03	Mercury	6.25	±	0.19
Sulfur	0.042	±	0.001	Nickel	20.6	±	1.1
Titanium	0.306	±	0.023	Selenium	1.52	±	0.14
				Silver	4.63	±	0.39
				Strontium	245.3	±	0.7
				Thallium	2.47	±	0.15
				Vanadium	81.6	±	2.9
				Zinc	350.4	±	4.8

NOTES

A3. Specifications

Handheld Analyzer	Description
Weight	3.35 lbs. (Base wt.), 3.70 lbs. (1.6 kg) with battery
Excitation Source	4 Watt X-ray tube - Au, Ag, Rh, or Ta anode (application optimized), 8-40 keV, 5 - 200 µA, eight filter positions
Detector	Various: Si PIN diode or Si Drift detector, thermo-electrically cooled, high resolution
Power	Removable Li-ion batteries, or AC power unit
Battery Life	Battery life varies depending on usage patterns
Display	Color "transflective" touchscreen (800 x 600) with 16-bit LCD interface)
Cal Check Coupon	316 Stainless Steel Alloy -- see also Docking Station
Power Requirements for AC Adapter	110-220 VAC, 50-60 Hz, 600 W max (P/N 100043)
Pressure Correction	Built-in barometer for automatic altitude correction
Operating Environment	Temperature: -10°C to 45°C Humidity: 10 to 90% Relative Humidity, non-condensing Altitude Rating: 2000 meters
Operating System	Windows Embedded CE®
Application Software	Innov-X Systems' proprietary Data Acquisition and Processing package.
USB Interface	USB 2.0

Operating specifications ar posted as **nominal.**

Docking Station	Description
Dimensions	L=14.0 in x W=8.75 in x H=4.75 in (H w/ spare battery = 6.5 in); Wt = 3.25 lbs.
Power Requirement	AC adapter standard: 110-220 VAC, 50-60 Hz, 600 W max
Cal Check Cup	Automatically performs Cal Check on inserted instrument
Battery Charging	Charges battery in unit; can simultaneously charge spare battery in separate socket

Accessories	Description
AC Battery Charger	P/N 120253
TestStand/Workstation	A-020-D - portable equipment stand making a fully shielded closed beam system. Uses Delta PC Software.
Soil Foot	A-035
Soil Extension Pole	P/N 990055
Trimble Xploration Package (P6000FDC)	**XPLORER FIELD DATA COLLECTION BUNDLE** **Trimble Nomad 800G-LC** - Ruggedized 800Mhz Field Computer with integrated GPS, Wi-Fi, Bluetooth, Camera, 16Gb Memory, SD Card Slot Options: Barcode Scanner, Cell Phone Interface, Fully DGPS compatible running Windows Mobile 6.0 OS.

Trimble Xploration Package (P6000TFG)	**XPLORER TOTAL FIELD GEOCHEM BUNDLE** Includes all features supplied with **Field Data Collection Package** and: **Soil Extension Pole** - Ergonomic extension pole facilitating soil analysis by a standing (and walking) operator. **Soil Foot** - Attaches to nose of DELTA analyzer balancing analyzer on ground for hands-free, extended in-situ testing. **ioGAS** - ioAnalytics GeoChemical Analysis Software Suite, an advanced spatial data analysis package for visualization, interrogation & validation of geochemical data for mineral exploration, mining, & environmental industries.

A4. Typical Delta Test Sequence

Innov-X Delta User Interface

Operators manage their measurement, analysis, and results activities from the *Innov-X* UI when in ANY of the Alloy modes/calibrations. This Appendix presents:

- Typical Sample Test Procedure, and
- Prerequisites for testing necessary for various modes

GO TO

The operational features of the ***Home, Mode, Setup, Test, and Results*** screens are explained in *"Delta User Interface Guide" (PN 103202)*
In this document, *"Delta User Manual,"* see *C4, Operations, Page 47* for a SNAP-SHOT of the UI.

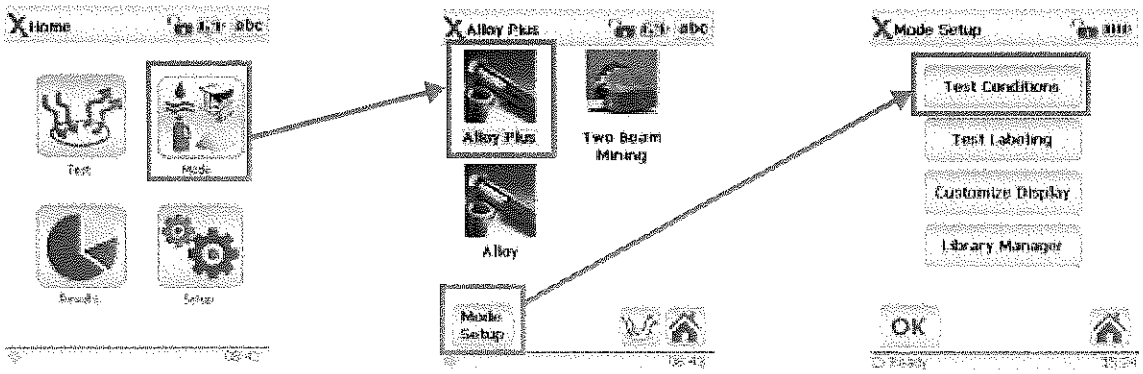
Typical Sample Test Procedure

This sample procedure features the Alloy Plus mode.

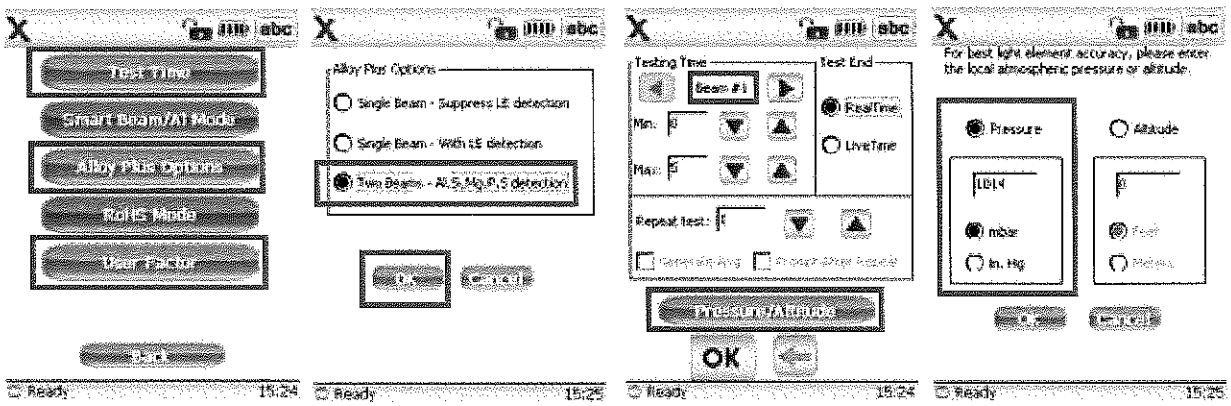
Other modes follow a similar sequence, however, the **Test Condition** screens are generally unique to each testing situation.

Use these steps:

1. From the **Home** screen, tap **Mode** icon
2. From **Mode** screen, tap the **Alloy Plus** icon
3. Select **Mode Setup** button



4. Select **Test Conditions**, then **Test Time**
5. Ensure that the **Testing Time** and other parameters are properly selected



6. Press OK button, the Back, then OK again
7. Return to Home screen, select **Test** to call the *Test-Alloy Plus* screen

Testing Tips for Certain Modes

Alloy:

- Place the analyzer's window on/over the test specimen, cover the window completely.
- Remember the Safety information (*C2, Pages 29-31*) to ensure your own personal safety.
- Take care not to damage the window film, such as when testing "metal turnings" or hot surfaces.

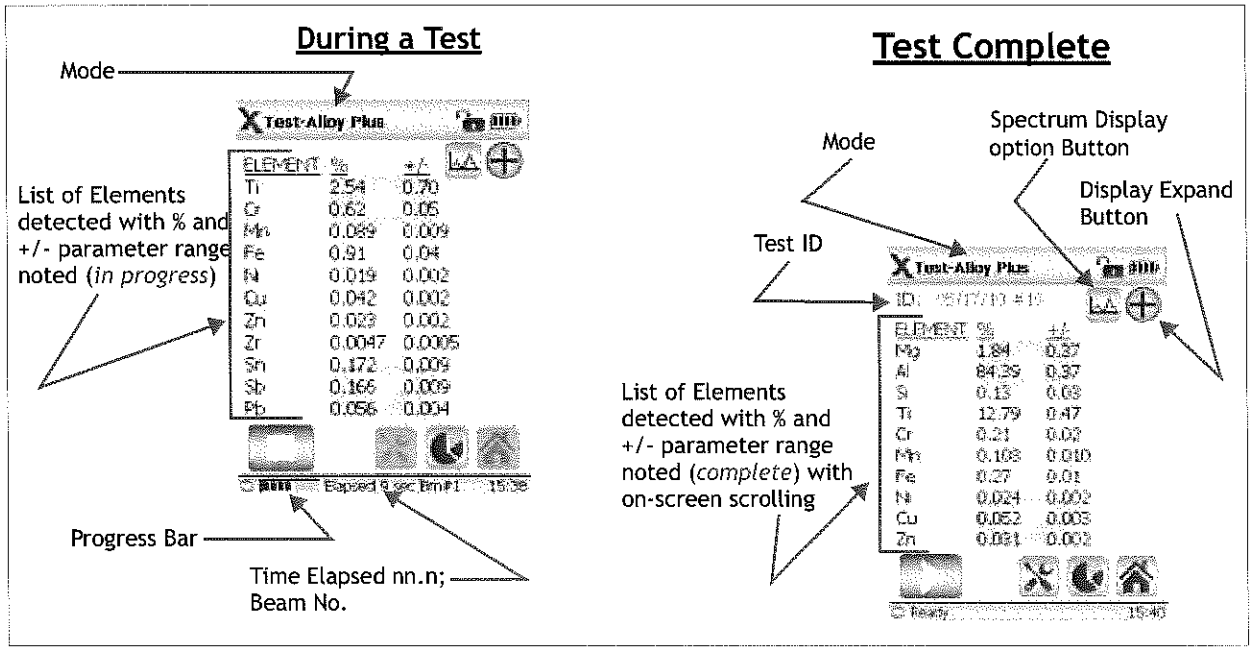
Soil or Mining:

- Place the analyzer's measurement window directly over the sample cup with the film side up.
- Specify the altitude or atmospheric pressure to gain increased accuracy for light elements.
- Confirm that the Testing Time and other parameters are properly selected
- Take care not to damage the window film, such as when testing uneven surfaces

RoHS:

- In order for XRF testing to be ***quantitative***, samples must be:
  - Homogeneous
  - Have a certain minimum sample thickness
    - Five (5) mm for polymers and light alloys
    - Fifteen (15) mm for liquid samples
    - One (1) mm for other alloys
- If samples are heterogeneous, too thin, or too small, only ***qualitative*** screening is possible.

8. Start a new test by using one of these methods:
   a. Tap **Start** button on the UI (toggles Delta to X-ray ON state)
   — or —
   b. Pull the trigger (toggles instrument to X-ray ON state)
   — or —
   c. Pull-and-hold the trigger when the "Deadman Trigger" is active.



Best Practices for Testing

Alloy Mode

Prior to a test session, analysts should have an understanding of *Innov-X*'s implementation of:

- **Grade Libraries** -> populated by alloy Grades -> their chemistry characterized by Grade Tables.
  See *Grade Libraries* for library management procedures.
- **Match Issues** including the concept and use of Match Numbers.
  See *Match Issues*

Prior to initiating testing with the analyzer software, **ensure** that the following tasks are complete:

1. Analyzer does not require a Cal Check procedure.
2. The analyzer's measurement window is properly positioned in front of or over the test sample.
3. The *Match Cutoff* and *nSigma* parameters are configured.

Mining and Soil Modes

Check Standards

- Measure a **check standard** after each Cal Check, and periodically throughout the day. Test for a recommended minimum of one minute.
- Concentrations for elements of interest, in the range expected, plus or minus the error on the reading, should be within 20 percent of the standard value.
- *A2.Soil Testing* describes recommended quality assurance details.

- The standards provided with the analyzer are contained in XRF sample cups. These containers have a window (through which the soil can be viewed) on one side, and a solid cap on the other side.

Always measure samples through the window.

Sample Presentation

IN SITU TESTING

In situ testing is performed by pointing the analyzer at the ground. Clear any grass or large rocks away and hold the analyzer with the probe head front flush to the ground. Since dirt can accumulate on the analyzer window, gently wipe the window clean after each analysis. Ensure the window is not ripped or punctured.

BAGGED OR PREPARED SAMPLE TESTING

Analyze prepared samples in a sample cup, through its window. Place the instrument's measurement window directly over the sample cup with the film side up.

Preparation considerations include:

- Avoid measuring very thin samples, as this can affect results. Prepare samples cups to contain at least 15 (usually 4-8 grams) mm of packed samples.
- When analyzing bagged samples, ensure that sufficient sample material exists in the bag to create a a sample thickness of a minimum of 15 mm for a spot size that is larger than the analyzer's measurement window.
- When using bags, cheaper bags (having thinner plastic walls) are better than more expensive ones (which have thicker plastic walls).

Consumer Mode

RoHS Best Practices

Check Standards

Innov-X Systems recommends that a check standard be measured after each Cal Check procedure, and periodically throughout the day.

Two certified standards are provided for verification.

- At least one standard should be measured for a minimum of two minutes.
- Concentrations for target elements (plus or minus the error on the reading) should be within 20% of the standard value.
- Standards provided are contained in XRF sample cups with a window (through which the plastic pellets can be viewed) on one side, and a solid cap on the other side.
- Samples should be measured in the sample cup, through the window.

Sample Presentation

Since many pieces of plastic analyzed for ROHS compliance are very small, take care to measure them in a safe and accurate manner.

See the IEC-ACEA recommendation for minimum thickness of test samples as shown in Chapter *C.8*.

A5. User Maintenance

5.1 Alternative Techniques for Powering or Charging the Delta

5.1.1. AC Power Adapter Kit

The kit is an **optional** accessory.

It is supplied with a 10 foot AC power cord, a switching AC adapter, and a Battery Module. This kit eliminates the Li-ion battery requirement, however the user is constrained by the length of the AC power cord.

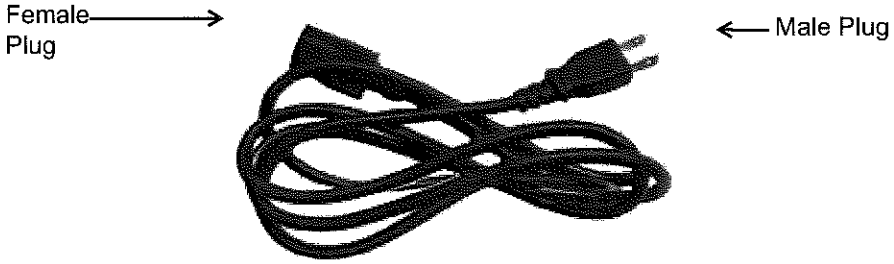
The procedure for using the AC adapter is shown below.

WARNING

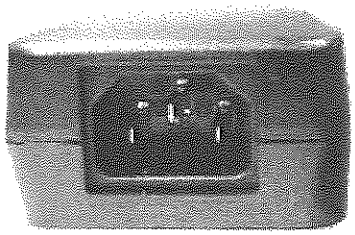
Ensure that each AC supply circuit has adequate power load capability and is provided from a grounded AC receptacle.

TO SETUP THE ADAPTER:

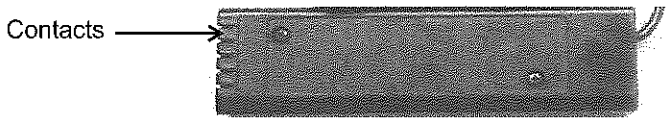
1. Plug the three-prong male plug into the receptacle.



2. Insert the three-prong female plug into the male receptacle on the *Switching AC Adaptor* brick.



3. Slide the Battery Eliminator unit into the analyzer with the contacts to the left. It is keyed to only go in oriented this way.



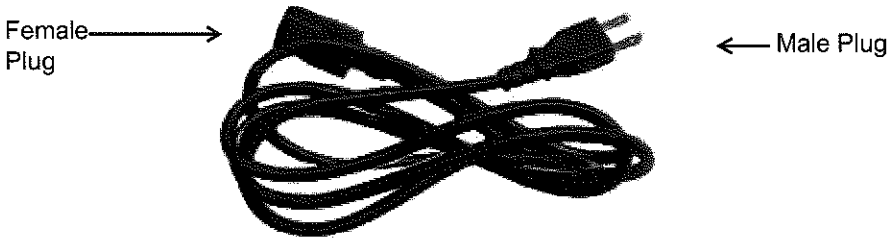
4. The user now has no limit to instrument power; not restricted by battery charge level. The range of action is constrained by the length of the power cord.

5.1.2. Li ion Battery Charger Assembly

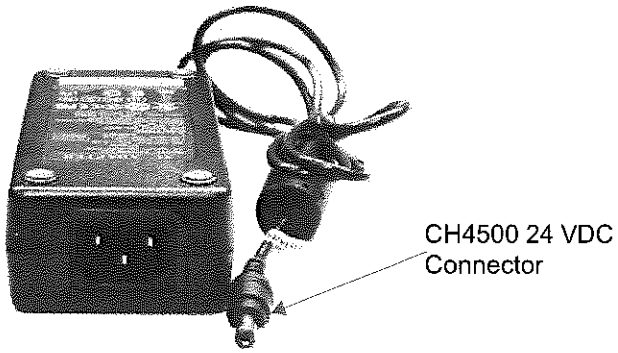
This section describes the procedure for charging the Li-ion batteries when the Delta Docking Station is NOT available. It features a single socket standalone battery charger (P/N 120253) The unit takes about two hours to completely charge a battery. Status of the battery's charge is shown by two lights on the power adaptor.

TO CHARGE THE BATTERY:

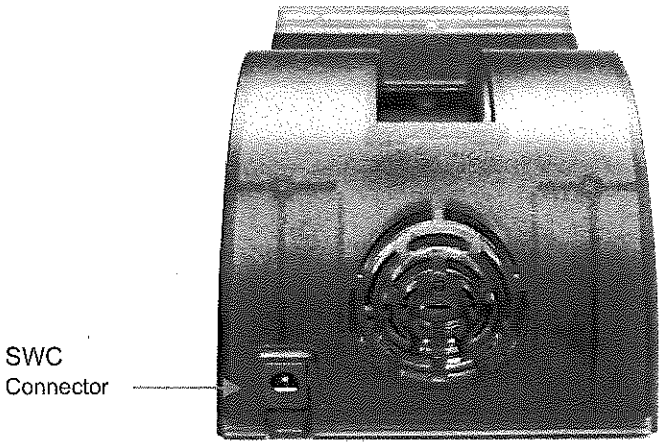
1. Plug the three-prong male plug into the Modes receptacle.



2. Plug the three-prong female plug into the male receptacle on the *AC Adaptor* brick.



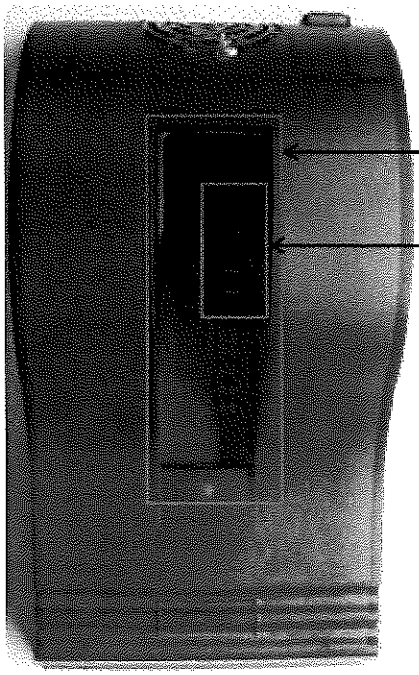
3. Plug the connector labeled *CH4500 24 VDC* into the connector labeled *SWC* on the back of the charger.



4. Insert the Li-ion battery into the charger with the contacts facing right.

CAUTION

Forcing the battery in with the contacts oriented improperly will injure the contacts and destroy the battery.



Battery Charger Status Lights

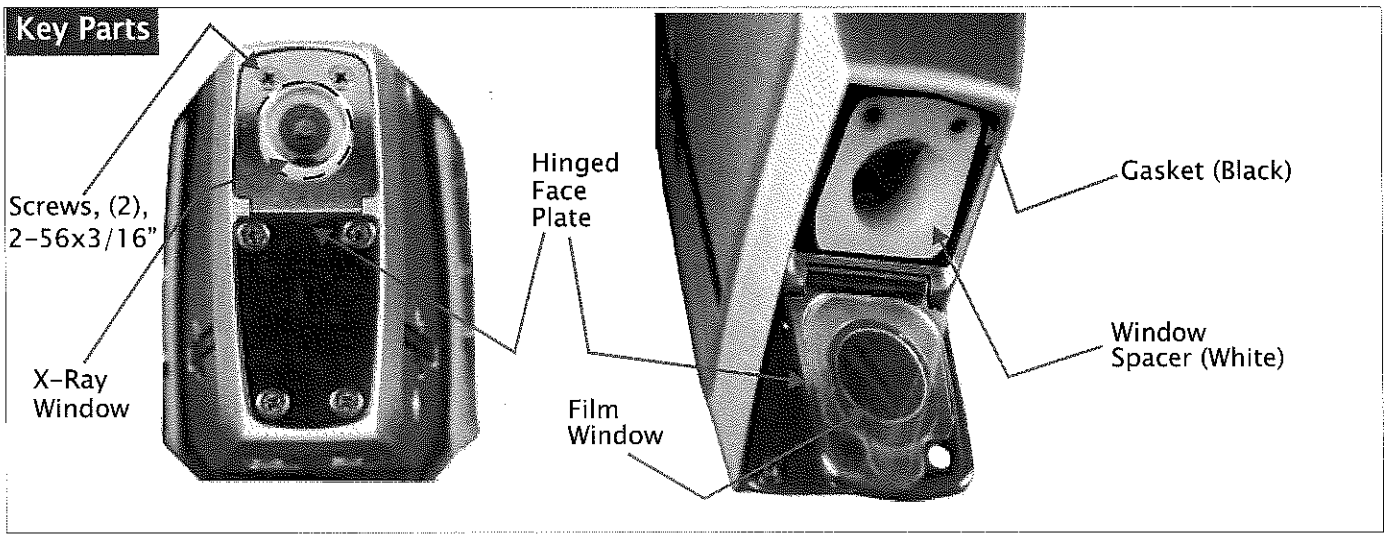
Left Light (Green only)	Right Light (Red only)	Status
On - Flashing	Off	Battery is charging
On - Solid	Off	Battery is charged
Off	On - Solid	Error. Remove battery and replace on charger. If the error persists, call Innov-X Systems Technical support.
Off	Off	No battery is on charger

5.2 — Window Replacement for "Hinged Plate" Analyzers

How to Replace the Window on a Delta Analyzer (All Models)

Tools: *Required* - Small Phillips Head Screwdriver
Recommended - Tweezers or Needle nose pliers; Can of compressed air
As needed: - Lint-free wipes or swabs

Key Parts



Remove Old Window

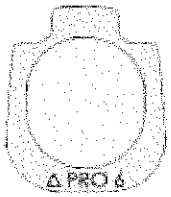
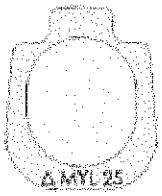
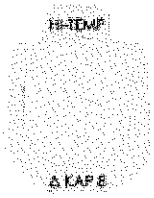
Use these guidelines:

- During this entire removal process, keep dust or foreign material out of the instrument.
- Ensure that the instrument is powered off.
- Take care to have clean hands.
- Orient the instrument with the nose pointing up.
- Review the **TIPS** section on next page before opening the hinged plate.
- Use Phillips head screwdriver to remove the two screws.
  - Carefully set aside the screws.
- Swing the hinged face plate out to expose the film window.
- Remove the old window.
- Observe the white plastic window spacer and the black rubber gasket
- Remove any dirt or foreign material from the gasket with compressed air
- Take EXTREME CARE to not harm any internal components

CAUTION **Avoid expensive internal damage, DO NOT INSERT anything (fingers or tools) into the instrument.**

Replacement Window Details

- Windows assemblies are made from either **Prolene, Mylar,** or **Kapton** film.
  - Users must specify the proper material to match their original application and calibration.
- The selected film is adhered to **one** side of a polyester backing plate; this creates a "front" and "back" surface
  - "Back" surface is completely covered with film.
  - "Front" surface shows a circular ridge where the film only is present, showing through the center hole of backing plate. This front surface also has a part type designator.
- Ensure that the "back" surface faces up when installing a new window (See below).

PRO 6 Prolene	MYL 25 Mylar	KAP 8 Kapton
		
P/N: –>> 102999	103000	103173 (Hi-Temp) 103115 (Classic Standard)

Install Replacement

Follow these steps:

- Remove new window assembly from shipping envelope.
- Handle it by edges or use tweezers.
- Apply canned compressed air to clear any contaminants from the surfaces.
- Place window assembly on the hinged plate with film back surface facing up.
  - Center it to avoid the screw holes.
- Carefully rotate the hinged plate up to secure the window assembly.
- Insert the two screws; tighten only to "snug".
  - Don't over-tighten them or strip the threads.

TIPS

- Do not touch the film in the center opening (on either side).
- When using the screwdriver, keep a finger or thumb between the tool and the X-ray window. This prevents accidentally sticking the tool through the film and causing internal damage.
- The cost for replacement windows is quite low. Users are encouraged to change them frequently in order to obtain optimum test results.
- When not operating an instrument, experienced users routinely keep it the Delta Docking Station cradle.
  - This keeps the window clean, and
  - Prevents accidental damage to the window film, and
  - Takes advantage of the battery charging and Cal Checking procedure that the DDS offers.

NOTES

A6. Packing and Shipping

If the instrument is not returned in the protective case, it can be damaged during shipping. Innov-X Systems reserves the right to void the warranty on instruments that are damaged during return shipping that are sent without the protective case.

Prior to returning a unit, contact Customer Service at the appropriate depot:

— United States —

- Phone: 1-781-938-5005
- Fax: 1-781-938-0128
- Email: Service@Innov-Xsys.com

- Mail & Shipping Address:
  Innov-X Systems, Inc.
  100 Sylvan Road
  Woburn, MA 01801

— Canada —

- Phone: 1-778-960-6279
- Fax: 1-604-568-2474
- Toll Free Fax: 1-888-873-6598
- Email: service@innovx.ca

- Mail & Shipping Address:
  Innov-X Canada
  1201 West Georgia, Ste. 2
  Vancouver BC
  Canada V6E 3J5

— Europe —

- Phone: +31 (0)73 62 72 590
- Fax: +31 (0)73 62 72 599
- Email: info@innovx-europe.com

- Mail & Shipping Address:
  Innov-X Systems, Inc.
  Kasteleinenkampweg 9R
  5222 AX 's-Hertogenbosch
  The Netherlands

— Australia —

- Phone: 02 9577 9500
- Fax: 02 9519 1850
- Email: service@innovx.com

- Mail & Shipping Address:
  Innov-X Systems Australia PTY LTD
  Suite 6, Level 3
  215 Euston Road
  Alexandria NSW 2015
  Australia

Or call your local distributor.

Ensure that you receive the required RMA number.

Follow these instructions to return your XRF Analyzer:

1. Pack the analyzer in the black protective case in which it arrived, using the original packing materials.
2. Include the RMA in the case and reference the RMA number in your shipping documents.
3. Close the protective case and either:
   - Secure it with plastic zip ties,
     — or —
   - Pack the protective case within another box.

Regulations for Shipping Products with Lithium Ion Batteries

The United States and many other countries have instituted regulations that require shippers to use a special Caution label referring to a Lithium Ion Battery.

- Label must be prominently displayed on the outer shipping container of any product that contains a Lithium Ion battery.
- Shipper may copy the label shown below to facilitate making the warning label. Use a color copier if possible.

A7. Legal Information

This section provides copies of the following:

- Delta Analyzer Limited Warranty including:
  - Limitation of Liability
  - Warranty Period, Returns, and Repairs
  - Instructions for Contacting Innov-X
- End User Software License Agreement including:
  - Use, Restrictions, and Termination of Software
  - Governmental End User Conditions
  - Limited Warranty and Limitation of Liability

Innov-X Delta Analyzer Limited Warranty

General Terms

EXCEPT AS EXPRESSLY SET FORTH IN THIS LIMITED WARRANTY, INNOV-X SYSTEMS, INC. (INNOV-X) MAKES NO OTHER WARRANTIES OR CONDITIONS, EXPRESSED OR IMPLIED, INCLUDING ANY IMPLIED WARRANTIES OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE. INNOV-X EXPRESSLY DISCLAIMS ALL WARRANTIES AND CONDITIONS NOT STATED IN THIS LIMITED WARRANTY. ANY IMPLIED WARRANTIES THAT MAY BE IMPOSED BY LAW ARE LIMITED IN DURATION TO THE LIMITED WARRANTY PERIOD.

This Limited Warranty applies to Innov-X analyzers sold or leased from Innov-X, its affiliates, authorized resellers, or country distributors (collectively referred to in this Limited Warranty as "Innov-X."

Innov-X warrants that the analyzer and all its internal and external components that you have purchased are free from defects in materials or workmanship under normal use during the Limited Warranty Period. The Limited Warranty Period starts on the date of shipment by Innov-X. You may be required to provide proof of purchase or lease as a condition of receiving warranty service. You are entitled to warranty service according to the terms and conditions of this document if a repair to your Innov-X analyzer is required within the Limited Warranty Period.

During the Limited Warranty Period, Innov-X will repair or replace the defective component parts. All component parts removed under this Limited Warranty become the property of Innov-X. In the unlikely event that your Innov-X analyzer has a recurring failure, Innov-X, at its discretion, may elect to provide you with a replacement unit of Innov-X's choosing that is at least equivalent to your Innov-X analyzer. This is your exclusive remedy for defective products. The repaired or replacement analyzer is warranted for the remainder of the limited Warranty Period.

YOU SHOULD MAKE PERIODIC BACKUP COPIES OF THE DATA STORED ON THE ANALYZER'S SYSTEM COMPUTER AS A PRECAUTION AGAINST POSSIBLE FAILURES, ALTERATION, OR LOSS OF THE DATA. BEFORE RETURNING ANY UNIT FOR SERVICE, BE SURE TO BACK UP DATA AND REMOVE ANY CONFIDENTIAL, PROPRIETARY, OR PERSONAL INFORMATION. INNOV-X IS NOT RESPONSIBLE FOR DAMAGE TO OR LOSS OF ANY PROGRAMS, OR DATA. INNOV-X IS NOT RESPONSIBLE FOR THE RESTORATION OR REINSTALLATION OF ANY PROGRAMS OR DATA OTHER THAN SOFTWARE INSTALLED BY INNOV-X WHEN THE ANALYZER IS MANUFACTURED.

Innov-X does not warrant that the operation of this analyzer will be uninterrupted or error-free. Innov-X is not responsible for damage that occurs as a result of your failure to follow the instructions that came with the Innov-X analyzer.

This Limited Warranty does not apply to expendable parts. This Limited Warranty does not extend to any analyzer from which the serial number has been removed or that has

been damaged or rendered defective (a) as a result of accident, misuse, abuse, or other external causes; (b) by operation outside the usage parameters stated in user documentation that shipped with the product; (c) by modification or service by anyone other than (i) Innov-X, or (ii) an Innov-X authorized service provider; (d) installation of software not approved by Innov-X.

These terms and conditions constitute the complete and exclusive warranty agreement between you and Innov-X regarding the Innov-X analyzer you have purchased or leased. These terms and conditions supersede any prior agreements or representations --- including representations made in Innov-X sales literature or advice given to you by Innov-X or any agent or employee of Innov-X --- that may have been made in connection with your purchase or lease of the Innov-X analyzer. No change to the conditions of this Limited Warranty is valid unless it is made in writing and signed by an authorized representative of Innov-X.

Limitation of Liability

IF YOUR INNOV-X ANALYZER FAILS TO WORK AS WARRANTED ABOVE, YOUR SOLE AND EXCLUSIVE REMEDY SHALL BE REPAIR OR REPLACEMENT. INNOV-X'S MAXIMUM LIABILITY UNDER THIS LIMITED WARRANTY IS EXPRESSLY LIMITED TO THE LESSER OF THE PRICE YOU HAVE PAID FOR THE ANALYZER OR THE COST OF REPAIR OR REPLACEMENT OF ANY COMPONENTS THAT MALFUNCTION IN CONDITION OF NORMAL USE.

INNOV-X IS NOT LIABLE FOR ANY DAMAGE CAUSED BY THE PRODUCT OR THE FAILURE OF THE PRODUCT TO PERFORM INCLUDING ANY LOST PROFITS OR SAVINGS OR SPECIAL, INCIDENTAL, OR CONSEQUENTIAL DAMAGES. INNOV-X IS NOT LIABLE FOR ANY CLAIM MADE BY A THIRD PARTY OR MADE BY YOU FOR A THIRD PARTY.

THIS LIMITATION OF LIABILITY APPLIES WHETHER DAMAGES ARE SOUGHT, OR A CLAIM MADE, UNDER THIS LIMITED WARRANTY OR AS A TORT CLAIM (INCLUDING NEGLIGENCE AND STRICT PRODUCT LIABILITY), A CONTRACT CLAIM, OR ANY OTHER CLAIM. THIS LIMITATION OF LIABILITY CANNOT BE WAIVED OR AMENDED BY ANY PERSON. THIS LIMITATION OF LIABILITY WILL BE EFFECTIVE EVEN IF YOU HAVE ADVISED INNOV-X OR AN AUTHORIZED REPRESENTATIVE OF INNOV-X OF THE POSSIBILITY OF ANY SUCH DAMAGES.

Software

This Limited Warranty does not warrant software products. The Innov-X software installed on the analyzer's system computer is covered by the Innov-X End User Software License Agreement.

Warranty Period

The warranty period for an Innov-X Delta Analyzer is one year. This warranty does not extend to expendable parts. Extended warranties are available from Innov-X.

Warranty Returns

A Return Material Authorization (RMA) Number must be obtained from the INNOV-X Service Department before any items can be shipped to the factory. Returned goods will not be accepted without an RMA Number. Customer will bear all shipping charges for warranty repairs. All goods returned to the factory for warranty repair should be properly packed to avoid damage and clearly marked with the RMA Number.

Warranty Repairs

Warranty repairs will be done either at the customer's site or at the INNOV-X plant, at our option. All service rendered by INNOV-X will be performed in a professional manner by qualified personnel.

Contacting Innov-X

Be sure to have the following information available before you call Innov-X:

- Analyzer serial number, model name, and model number
- Applicable error messages
- Description of problem
- Detailed questions

Methods of Contact

— United States —

- Phone: 1-781-938-5005
- Fax: 1-781-938-0128
- Email: Service@Innov-Xsys.com

- Mail & Shipping Address: Innov-X Systems, Inc. 100 Sylvan Road Woburn, MA 01801

— Canada —

- Phone: 1-778-960-6279
- Fax: 1-604-568-2474
- Toll Free FAX: 1-888-873-6598
- Email: service@innovx.ca

- Mail & Shipping Address: Innov-X Canada 1201 West Georgia, Ste. 2 Vancouver BC Canada V6E 3J5

— Europe —

- Phone: +31 (0)73-62 72 590
- Fax: +31 (0)73-62 72 599
- Email: info@innovx-europe.com

- Mail & Shipping Address: Innov-X Systems, Inc. Kasteleinenkampweg 9R 5222 AX 's-Hertogenbosch The Netherlands

— Australia —

- Phone: 02 9577 9500
- Fax: 02 9519 1850
- Email: service@innovx.com

- Mail & Shipping Address: Innov-X Systems Australia PTY LTD 215 Euston Road, # 6 / L3 Alexandria, NSW, 2015 Australia

Or call your local distributor.

End User Software License Agreement

THIS END USER SOFTWARE LICENSE AGREEMENT IS FOR THE SOFTWARE USED TO OPERATE THE INNOV-X SYSTEMS' Delta™ XRF ANALYZER AND LIMITED PRODUCT WARRANTY

NOTICE TO USER:

PLEASE READ THIS DOCUMENT CAREFULLY. THIS IS THE CONTRACT BETWEEN YOU AND INNOV-X SYSTEMS, INC. (INNOV-X), REGARDING THE OPERATING SOFTWARE FOR YOUR INNOV-X Delta™ XRF ANALYZER INSTRUMENT.

THIS LICENSE COVERS THE INNOV-X SOFWARE INSTALLED ON THE DELTA™ XRF ANALYZER'S SYSTEM COMPUTER AND THE INNOV-X SOFTWARE OR FIRMWARE INSTALLED ON THE COMPUTER CHIPS WITHIN YOUR ANALYZER ENCLOSURE. THIS AGREEMENT CONTAINS WARRANTY AND LIABILITY DISCLAIMERS AND LIMITATIONS. YOUR INSTALLATION AND USE OF THE INNOV-X SOFTWARE IS SUBJECT TO THE TERMS AND CONDITIONS CONTAINED IN THIS END USER SOFTWARE LICENSE AGREEMENT. THIS LICENSE DOES NOT COVER THE OPERATING SYSTEM SOFTWARE AND OTHER SOFTWARE THAT MAY BE INCLUDED.

This Innov-X Systems, Inc. **End User Software License Agreement** accompanies an Innov-X Systems, Inc. Analyzer (including the external System Computer) with the software product installed ("Software") and related explanatory materials ("Documentation"). The term "Software" also includes any upgrades, modified versions, updates, additions and copies of the Software licensed to you by Innov-X Systems, Inc. The term "License" or "Agreement" means this End User Software License Agreement. The term "you" or "Licensee" means the purchaser of this license to use the Software.

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Use of the Software

1. Subject to the terms and conditions of this Agreement, Innov-X Systems, Inc. grants the purchaser of this product a non-exclusive license only to use the Software installed on the system computer that is integrated with your Analyzer and to use the software installed on the circuit boards that are installed in your Innov-X analyzer.
2. You may make one copy of the Software in machine-readable form solely for backup or archival purposes. You must reproduce on any such copy all copyright notices and any other proprietary legends found on the original. You may not make any other copies of the Software.

Restrictions

1. You may not copy, transfer, rent, modify, use or merge the Software, or the associated documentation, in whole or in part, except as expressly permitted in this Agreement.
2. You may not reverse assemble, decompile, or otherwise reverse engineer the Software.
3. You may not remove any proprietary, copyright, trade secret or warning legend from the Software or any Documentation.
4. You agree to comply fully with all export laws and restrictions and regulations of the United States or applicable foreign agencies or authorities. You agree that you will not export or re-export, directly or indirectly, the Software into any country prohibited by the United States Export Administration Act and the regulations there under or other applicable United States law.
5. You may not modify, sell, rent, transfer (except temporarily in the event of a computer malfunction), resell for profit, or distribute this license or the Software, or create derivative works based on the Software, or any part thereof or any interest therein. Notwithstanding the foregoing, you may transfer this Software to a purchaser of the specific Innov-X Analyzer in or for which this Software is installed in connection with any sale of such Analyzer, provided that the transferee agrees to be bound by and to comply with the provisions of this Agreement. A re-licensing fee may be charged for any such replacement software.

Termination

You may terminate this Agreement by discontinuing use of the Software, removing all copies from the system computer' hard drive and returning the Software and Documentation, and all copies thereof, to Innov-X Systems, Inc. Innov-X Systems, Inc. may terminate this Agreement if you fail to comply with all of its terms, in which case you agree to discontinue using the Software, remove all copies from the system computer and storage media, and return all copies of the Documentation thereof, to Innov-X Systems, Inc.

WARNING: Although it is theoretically possible for you to write software to operate the Innov-X analyzer, you are required to return the software as a result of failing to comply with the terms of this license, it is likely that the Analyzer purchased with the software covered by this License will not work properly. In addition, any remaining term under the warranty on the Analyzer will be voided.

U.S. Government End Users

The Software is a "commercial item," as that term is defined in 48 C.F.R. 2.101 (Oct. 2006 ED.), consisting of "commercial computer software" and "commercial computer software documentation," as such terms are used in 48 C.F.R. 12.212 (Oct. 2006 ED). Consistent with 48 C.F.R. 12.212 and 48 C.F.R. 227.7202-1 through 227.7202-4 (Oct. 2006 ED.), all U.S. Government End Users acquire the Software with only those rights set forth herein.

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If this Software is used within a country of the European Community, nothing in this Agreement shall be construed as restricting any rights available under the European Community Software Directive 2006/116/EC.O.J. Published in (L.372) V49 (27 Dec. 2006).

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Limited warranty and Limitation of remedies

Limited Warranty

Innov-X Systems, Inc. warrants that for a period of ninety days from the beginning of the applicable warranty period (as described below), or for the designated warranty period if a different warranty period is designed as the warranty period for the Software in the current version of an instrument operating manual or catalog or in a specific written warranty including with and covering the Software, the Software will function substantially in accordance with the functions and features described in the Documentation delivered with the Software when properly installed.

The above warranties do not apply to defects resulting from misuse, neglect, or accident, including without limitation: operation outside of the Innov-X analyzer or use specifications, or not in conformance with the instructions for any instrument system, software, or accessories; improper or inadequate maintenance by the user; installation of software or interfacing, or use in combination with software or products not supplied or authorized by Innov-X Systems, Inc.; and modification or repair of the analyzer not authorized by Innov-X Systems, Inc.

Warranty Period Commencement Date.

The applicable warranty period for software begins on the earlier of the date of installation or three (3) months from the date of shipment for software installed by Innov-X Systems, Inc.' personnel. For software installed by the purchaser or anyone other than Innov-X Systems, Inc., the warranty period begins on the date the software is delivered to you. The applicable warranty period for media begins on the date the media is delivered to the purchaser.

INNOV-X SYSTEMS, INC. MAKES NO OTHER WARRANTIES OF ANY KIND WHATSOEVER, EXPRESS OR IMPLIED, WITH RESPECT TO THE SOFTWARE OR DOCUMENTATION, INCLUDING BUT NOT LIMITED TO WARRANTIES OF FITNESS FOR A PARTICULAR PURPOSE OR MERCHANTABILITY OR THAT THE SOFTWARE OR DOCUMENTATION IS NON-INFRINGING. ALL OTHER WARRANTIES ARE EXPRESSLY DISCLAIMED. WITHOUT LIMITING THE GENERALITY OF THE FOREGOING, INNOV-X SYSTEMS, INC. MAKES NO WARRANTIES THAT THE SOFTWARE WILL MEET YOUR REQUIREMENTS, THAT OPERATION OF THE LICENSED SOFTWARE WILL BE UNINTERRUPTED OR ERROR FREE OR WILL CONFORM EXACTLY TO THE DOCUMENTATION, OR THAT INNOV-X SYSTEMS, INC. WILL CORRECT ALL PROGRAM ERRORS. INNOV-X SYSTEMS, INC.' SOLE LIABILITY AND RESPONSIBILITY FOR BREACH OF WARRANTY RELATING TO THE SOFTWARE OR DOCUMENTATION SHALL BE LIMITED, AT INNOV-X SYSTEMS, INC.' SOLE OPTION, TO (1) CORRECTION OF ANY ERROR IDENTIFIED TO INNOV-X SYSTEMS, INC. IN WRITING FROM YOU IN A SUBSEQUENT RELEASE OF THE SOFTWARE, WHICH SHALL BE SUPPLIED TO YOU FREE OF CHARGE, (2) REPLACEMENT OF THE DEFECTIVE SOFTWARE WITH A FUNCTIONALLY EQUIVALENT PROGRAM AT NO CHARGE TO YOU, OR (3) PROVIDING A REASONABLE WORK AROUND WITHIN A REASONABLE TIME. INNOV-X SYSTEMS, INC. SOLE LIABILITY AND RESPONSIBILITY UNDER THIS AGREEMENT FOR BREACH OF WARRANTY RELATING TO MEDIA IS THE REPLACEMENT OF DEFECTIVE MEDIA RETURNED WITHIN 90 DAYS OF THE DELIVERY DATE. THESE ARE YOUR SOLE AND EXCLUSIVE REMEDIES FOR ANY BREACH OF WARRANTY. WARRANTY CLAIMS MUST BE MADE WITHIN THE APPLICABLE WARRANTY PERIOD.

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General

This Agreement shall be governed by laws of the State of Massachusetts, exclusive of its conflict of laws provisions. This Agreement shall not be governed by the United Nations Convention on Contracts for the International Sale of Goods. This Agreement contains the complete agreement between the parties with respect to the subject matter hereof, and supersedes all prior or contemporaneous agreements or understandings, whether oral or written. If any provision of this Agreement is held by a court of competent jurisdiction to be contrary to law that provision will be enforced to the maximum extent permissible and the remaining provisions of this Agreement will remain in full force and effect. The controlling language of this Agreement, and any proceedings relating to this Agreement, shall be English. You agree to bear any and all costs of translation, if necessary. The headings to the sections of this Agreement are used for convenience only and shall have no substantive meaning. All questions concerning this Agreement shall be directed to Innov-X Systems, Inc., 100 Sylvan Road, Suite 500, Woburn, MA 01801 USA. Tel: 1-781-938-5005 Fax: 1-781-938-0128

A8. Alloy Grade Libraries

Every Delta unit is supplied with four libraries:

1. "Factory" Library unique to every Model
   - See following pages for tables showing Model/Factory grade names
2. Tramp Library
3. User Library #1 (user may store more than 500 grade names)
4. User Library # 2 (user may store more than 500 grade names)

Libraries are editable. However, InnovX does not recommend that users edit the "Factory" grade library.

Tramp Library

Every analyzer is shipped with a "Tramp" library comprised of seven base alloys. The "Tramp" library supports other grade libraries.

Users can set Tramp Limits, element by element, alloy base by alloy base, to meet their specific requirements.

A single click can select/deselect (globally) the Tramp feature.

How the Tramp Library works:

1. Tramp Grades are matched to alloy bases rather than specific grades.
   - Each sample is determined to be one of seven possible base alloys - see list below
   - Analyzer applies the Tramp Grade/base specific tramp limits from the matching Tramp Grade;
2. These "Tramp Limits" or "alloy base specific" limits are applied when an element is detected in a specific grade.
   - But, the nearest grade match has no specification for that element,
   - And, the concentration of the sample is less than the max limit specified by the matching Tramp Grade.
3. When conditions of #2 are met, the element is reported on the User Interface screen.
   - In blue;
   - Is labeled as a "Tramp" material in the grade comparison table;
   - But the grade match is not penalized.

PRACTICAL ADVANTAGES of this "tramp element" approach:

- Faster sorting,
- Fewer ambiguous or incorrect matches,
- Improved grade library integrity,
- Prominent labeling of tramp elements.

TRAMP Library Base Alloys

_AlAlloyBase
Common Tramp Elements: Pb, Bi, Sn, Fe, Cu, Zn

_CoAlloyBase
Common Tramp Elements: Al, Ti, V, Cu, Nb, Ta, Zr

_CuAlloyBase
Common Tramp Elements: S, As, Ag, Sb, Sn; not as common Pb, Co, Ni

_FeAlloyBase
Common Tramp Elements: V, Co, Cu, Ni, As - sometimes Si, W, Nb

_GenericAlloyBase
Common Tramp Elements: V, Co, Cu, Ni, As - sometimes Si, W, Nb

_NiAlloyBase
Common Tramp Elements: V, Co, W, Zr, Nb - sometimes Ta, Mo, Cr, Cu

_TiAlloyBase
Common Tramp Elements: Fe is common, Cu & Si show up at low levels.

Factory Grade Library {Table A9.1-Classic}

Aluminums-C

2007
2011
2018
2117
2618
4032
5454
6040
6061
6070
6253
6262
7005
7016
7019
7039
7050
7072
7075
7104
1100-plus
2024-plus
2098-2195
2219-2519
3003 or 4 or 5
355-2
5052-plus
5086-plus
6063-plus
7049-149-249

Cobalt Alloys - C

Alloy 686
AlnicoVIII
Cobalt
Elgiloy
F75
FSX-414
Haynes188
Haynes36
HS-1
HS-12
HS-19
HS-21
HS25-L605
HS-31
HS-4
HS-6B
Jetalloy
MarM302
MarM509
MarM905
MP35N
MPN159
Star J
Ultimet

Specialty Grades- C

60Sn-40Pb
63Sn-37Pb
96-4
AZ31B
AZ91A or C
SAC 300
SAC 305
SAC 400
SAC 405
97-3
Ag
Au
Bi
Cb 103
CP Ta
Cr
Densalloy
Hf
Mn
Mo
Nb
Ni
Pb
Pd
Re
Sb
Se
Sn
TungCarb C
TungCarb S
V
W
Zn
Zr
Zr 2 or 4
Zr 702
Zr 704
Zr 705

Copper Alloys -C

C 110	C 510	C 864
C 172	C 524	C 867
C 194	C 534	C 868
C 210	C 544	C 875
C 220	C 623	C 8932
C 260	C 630	C 903
C 270	C 655	C 922
C 310	C 667	C 932
C 314	C 673	C 937
C 330	C 675	C 955
C 332	C 706	C194HiCu
C 340	C 710	C197HiCu
C 342	C 715	Elec Cu
C 360	C 745	Muntz
C 377	C 752	NarloyZ
C 425	C 814	SeBiLOYI
C 443	C 836	SeBiLOYII
C 464	C 857	SeBiLOYIII
C 482		
C 485		

Nickel Alloys - C

B 1900	I-617	MarM421
B-1900 Hf	I-625	Monel400
C-1023	I-690	Monel411
GMR235	I-700	MonelK500
GTD222	I-702	MuMetal
Hast BC1	I-706	Ni 200
HastB	I-713	NichromeV
HastB2	I-718	Nim101
HastB3	I-720	Nim263
HastC2000	I-722	Nimonic75
HastC22	I-725	Nimonic80A
HastC276	I-738	Nimonic90
HastC4	I-750	PWA1480
HastF	I-792	PWA1484
HastG	I-800	RA333
HastG2	I-801	Rene125
HastG3	I-825	Rene142
HastG30	I-901	Rene220
HastN	I-903	Rene41
HastR	I-907-909	Rene77
HastS	I-939	Rene80
HastW	IN100Mar	Rene95
HastX	M002	Supertherm
Haynes230	MarM200	Udimet500
HR160	MarM246	Udimet520
HyMu80	MarM247	Udimet700
I-102I-49		Waspaloy
I-600		
I-601		

Low Alloy Steels-C

3310
4130
4140
4340
8620
9310
12L14
A10
Carb 1-2
Moly
Carbon
Steel
20Mo4

Chrome-Moly Steels -C

1 1-4 Cr
2 1-4 Cr
5 Cr
9 Cr
P91

Factory Grade Library {Table A9.2-Classic}

Stainless Grades - C		
201	21-6-9	Haynes556
203	25-4-4	Incoloy840
304	254SMO	Invar 36
309	26-1	Kovar
310	29-4	M152
316	29-4-2	Maraging350
317	29-4C	MaragingC200
321	302HQ	MaragingC250
329	410 Cb	MaragingC300
330	410-16-20	N-155
347	904L	Ni-hard#1
422	A-286	Ni-hard#4
430	AL6XN	Ni-Span902
431	Alloy42	Nitronic40
434	AlnicoII	Nitronic50
440	AlnicoIII	Nitronic60
441	AlnicoV	RA330
446	AMS350	RA85H
2003	AMS355	Zeron100
2101	CD4MCU	
2507	Custom450	
13-8 Mo	Custom455	
15-5 PH	Duplex2205	
15Mn7Cr	E-bite	
17-4 PH	Ferallium255	
17-7 PH	GreekAs-coloy	
19-9DL	H12	
19-9DX	H13	
20Cb3		
20Mo6		

Ti Grades - C
Cp Ti
Cp Ti Pd
Ti 12
Ti 17
Ti 3 2-5
Ti 6-22-22
Ti 6-2-4-2
Ti 6-2-4-6
Ti 6-4
Ti 6-6-2
Ti 8
Ti 8-1-1
Ti10-2-3
Ti15-3-3-3
Ti3-11-13
Ti5 - 2-5
Ti6-2-1-1
TiBetaC

Tool Steels- C
A2
A6
A7
D2 or D4
D7
H-11
M1
M2
M4
M42
O1
O2
O6
O7
S1
S5
S6
S7
T1

Factory Grade Library {Table A9.3-Standard}

Delta STANDARD

Aluminums-S

319
333
380
383
384
2007
2011
2018
2024
2117
2618
3004
4032
5042
5052
5083
5086
5154
5454
6040
6061
6070
6253
6262
7005
7016
7019
7039
7050
7072
7075
7104
1100-plus
2014-17 std
2024-plus
2098-2195
2219-2519
3003 or 4 or 5
3003 or 5
355-2
356-57-std
5052-plus
5056-82
5086-plus
6063-plus
7049-149-249

Cobalt Alloys - S

Alloy 686
AlnicoVIII
Cobalt
Elgiloy
F75
FSX-414
Haynes188
Haynes36
HS-1
HS-12
HS-19
HS-21
HS25-L605
HS-31
HS-4
HS-6B
Jetalloy
MarM302
MarM509
MarM905
MP35N
MPN159
Star J
Ultimet

Low Alloy Steels-S

3310
4130
4140
4340
8620
9310
12L14
A10
Carb 1-2
Moly
Carbon Steel
P20
135 N

Specialty Grades- S

60Sn-40Pb
63Sn-37Pb
96-4
AZ31B
AZ91A or C
SAC 300
SAC 305
SAC 400
SAC 405
97-3
Ag
Au
Bi
Cb 103
CP Ta
Cr
Densalloy
Hf
Mn
Mo
Nb
Ni
Pb
Pd
Re
Sb
Se
Sn
TungCarb C
TungCarb S
V
W
Zn
Zr
Zr 2 or 4
Zr 702
Zr 704
Zr 705

Chrome-Moly Steels -S

1 1-4 Cr
2 1-4 Cr
5 Cr
9 Cr
P91

Copper Alloys -S

C 110	C 510	C 864
C 172	C 524	C 867
C 194	C 534	C 868
C 210	C 544	C 875
C 220	C 623	C 8932
C 260	C 630	C 903
C 270	C 655	C 922
C 310	C 667	C 932
C 314	C 673	C 937
C 330	C 675	C 955
C 332	C 706	C194HiCu
C 340	C 710	C197HiCu
C 342	C 715	Elec Cu
C 360	C 745	Muntz
C 377	C 752	NarloyZ
C 425	C 814	SeBiLOYI
C 443	C 836	SeBiLOYII
C 464	C 857	SeBiLOYIII
C 482		
C 485		

Nickel Alloys - S

20Mo4	I-102	MarM247
B 1900	I-49	MarM421
B-1900 Hf	I-600	Monel400
C-1023	I-601	Monel411
Colmonoy	I-602	MonelK500
6	I-617	MuMetal
GMR235	I-625	Ni 200
GTD222	I-690	NichromeV
Hast BC1	I-700	Nim101
HastB	I-702	Nim263
HastB2	I-706	Nimonic75
HastB3	I-713	Nimonic80A
HastC2000	I-718	Nimonic90
HastC22	I-720	PWA1480
HastC276	I-722	PWA1484
HastC4	I-725	RA333
HastF	I-738	Rene125
HastG	I-750	Rene142
HastG2	I-792	Rene220
HastG3	I-800	Rene41
HastG30	I-801	Rene77
HastN	I-825	Rene80
HastR	I-901	Rene95
HastS	I-903	Supertherm
HastW	I-907-909	Udimet500
HastX	I-939	Udimet520
Haynes214	IN100	Udimet700
Haynes230	MarM002	Waspaloy
HR160	MarM200	
HyMu80	MarM246	

Factory Grade Library {Table A9.4-Standard}

Stainless Grades - S		
201	17-4 PH	H12
203	17-7 PH	H13
303	19-9DL	Haynes556
304	19-9DX	Incoloy840
309	20Cb3	Invar 36
310	20Mo6	Kovar
316		M152
317	21-6-9	Maraging350
321	25-4-4	MaragingC200
329	254SMO	MaragingC250
330	26-1	MaragingC300
347	29-4	
410	29-4-2	N-155
416	29-4C	Ni-hard#1
420	302HQ	Ni-hard#4
422	410 Cb	Ni-Span902
430	410-16-20	Nitronic40
431	904L	Nitronic50
434	A-286	Nitronic60
440	AL6XN	RA330
441	Alloy42	RA85H
446	AlnicoII	Zeron100
2003	AlnicoIII	
2101	AlnicoV	
2205	AMS350	
2205	AMS355	
2507	CD4MCU	
13-8 Mo	Custom450	
15-5 PH	Custom455	
15Mn7Cr	E-bite	
	Ferallium255	
	GreekAscoloy	

Ti Grades - S
Cp Ti
Cp Ti Pd
Ti 12
Ti 17
Ti 3 2-5
Ti 6-22-22
Ti 6-2-4-2
Ti 6-2-4-6
Ti 6-4
Ti 6-6-2
Ti 8
Ti 8-1-1
Ti10-2-3
Ti15-3-3-3
Ti3-11-13
Ti5 - 2-5
Ti6-2-1-1
TiBetaC

Tool Steels- S
A2
A6
A7
D2 or D4
D7
H-11
M1
M2
M4
M42
O1
O2
O6
O7
S1
S5
S6
S7
T1

Factory Grade Library {Table A9.5-Premium}

Delta PREMIUM

Aluminums-P

319
333
356
357
380
383
384
1100
2007
2011
2018
2024
2117
2618
3002
3003
3004
3005
3105
4032
5005
5042
5052
5083
5086
5154
5454
5657
6040
6061
6063
6070
6253
6262
7005
7016
7019
7039
7050
7072
7075
7104
1100-plus
2014-17
2024-plus
2098-2195
2219-2519
3003 or 4 or 5
355-2
5052-plus
5056-82
5086-plus
6063-plus
7049-149-249

Cobalt Alloys - P

Alloy 686
AlnicoVIII
Cobalt
Elgiloy
F75
FSX-414
Haynes188
Haynes36
HS-1
HS-12
HS-19
HS-21
HS25-L605
HS-31
HS-4
HS-6B
Jetalloy
MarM302
MarM509
MarM905
MP35N
MPN159
Star J
Ultimet

Low Alloy Steels-P

3310
4130
4140
4340
8620
9310
12L14
A10
Carb 1-2
Moly
Carbon Steel
P20
135 N

Specialty Grades- P

60Sn-40Pb
63Sn-37Pb
96-4
AZ31B
AZ91A or C
SAC 300
SAC 305
SAC 400
SAC 405
97-3
Ag
Au
Bi
Cb 103
CP Ta
Cr
Densalloy
Hf
Mn
Mo
Nb
Ni
Pb
Pd
Re
Sb
Se
Sn
TungCarb C
TungCarb S
V
W
Zn
Zr
Zr 2 or 4
Zr 702
Zr 704
Zr 705

Chrome-Moly Steels -P

1 1-4 Cr
2 1-4 Cr
5 Cr
9 Cr
P91

Copper Alloys -P

C 110	C 510	C 863
C 172	C 524	C 864
C 194	C 534	C 867
C 210	C 544	C 868
C 220	C 623	C 875
C 240	C 630	C 8932
C 260	C 642	C 903
C 270	C 655	C 922
C 310	C 667	C 932
C 314	C 673	C 937
C 330	C 675	C 954
C 332	C 687	C 955
C 340	C 706	C194HiCu
C 342	C 710	C197HiCu
C 360	C 715	Elec Cu
C 377	C 745	Muntz
C 425	C 752	NarloyZ
C 443	C 814	SeBiLOYI
C 464	C 836	SeBiLOYII
C 482	C 857	SeBiLOYIII
C 485	C 861	

Nickel Alloys - P

20Mo4	I-102	MarM246
B 1900	I-49	MarM247
B-1900 Hf	I-600	MarM421
C-1023	I-601	Monel400
Colmonoy	I-602	Monel411
6	I-617	MonelK500
GMR235	I-625	MuMetal
GTD222	I-690	Ni 200
Hast BC1	I-700	NichromeV
HastB	I-702	Nim101
HastB2	I-706	Nim263
HastB3	I-713	Nimonic75
HastC2000	I-718	Nimonic80A
HastC22	I-720	Nimonic90
HastC276	I-722	PWA1480
HastC4	I-725	PWA1484
HastF	I-738	RA333
HastG	I-750	Rene125
HastG2	I-792	Rene142
HastG3	I-800	Rene220
HastG30	I-801	Rene41
HastN	I-825	Rene77
HastR	I-901	Rene80
HastS	I-903	Rene95
HastW	I-907-909	Supertherm
HastX	I-939	Udimet500
Haynes214	IN100	Udimet520
Haynes230	MarM002	Udimet700
HR160	MarM200	Waspaloy
HyMu80		

Factory Grade Library {Table A9.6-Premium}

Stainless Grades - P		
201	17-4 PH	H12
203	17-7 PH	H13
303	19-9DL	Haynes556
304	19-9DX	Incoloy840
309	20Cb3	Invar 36
310	20Mo6	Kovar
316	21-6-9	M152
317	25-4-4	Maraging350
321	254SMO	MaragingC200
329	26-1	MaragingC250
330	29-4	MaragingC300
347	29-4-2	N-155
410	29-4C	Ni-hard#1
416	302HQ	Ni-hard#4
420	410 Cb	Ni-Span902
422	410-16-20	Nitronic40
430	904L	Nitronic50
431	A-286	Nitronic60
434	AL6XN	RA330
440	Alloy42	RA85H
441	AlnicoII	Zeron100
446	AlnicoIII	
2003	AlnicoV	
2101	AMS350	
2205	AMS355	
2205	CD4MCU	
2507	Custom450	
13-8 Mo	Custom455	
15-5 PH	E-bite	
15Mn7Cr	Ferallium255	
	GreekAscoloy	

Ti Grades - P
Cp Ti
Cp Ti Pd
Ti 12
Ti 17
Ti 3 2-5
Ti 6-22-22
Ti 6-2-4-2
Ti 6-2-4-6
Ti 6-4
Ti 6-6-2
Ti 8
Ti 8-1-1
Ti10-2-3
Ti15-3-3-3
Ti3-11-13
Ti5 - 2-5
Ti6-2-1-1
TiBetaC

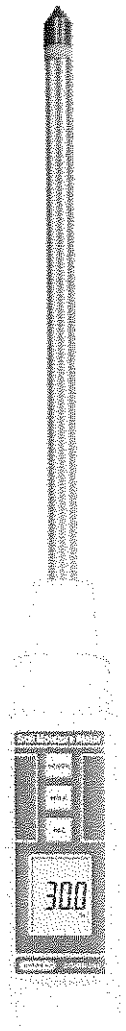
Tool Steels- P
A2
A6
A7
D2 or D4
D7
H-11
M1
M2
M4
M42
O1
O2
O6
O7
S1
S5
S6
S7
T1

GO TO

- See *"Delta User Interface Guide"* (PN 103202) for a complete description for using Alloy modes' new capabilities such as:
  - Grade Match Messaging,
  - SmartSorting,
  - Nominal Chemistry, and
  - Tramp Library management.

SOIL MOISTURE METER

Model : DSMM500



Your purchase of this SOIL MOISTURE METER marks a step forward for you into the field of precision measurement. Although this METER is a complex and delicate instrument, its durable structure will allow many years of use if proper operating techniques are developed. Please read the following instructions carefully and always keep this manual within easy reach.

OPERATION MANUAL

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2. SPECIFICATIONS.....1

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   3-2 Moisture Sensing Head.....3
   3-3 Power Button.....3
   3-4 Hold Button.....3
   3-5 REC Button.....3
   3-6 LCD Display.....3
   3-7 Battery Compartment/Cover.....3

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   4-1 Measurement by selecting the different material.....4
   4-2 Data Hold.....5
   4-3 Data Record.....5

5. BATTERY REPLACEMENT.....6

1. FEATURES

* Designed to check the moisture level of soil or other similar material.
* Measurement range : 0 % to 50 % moisture content of soil sample with 0.1 % resolution.
* All in one digital soil meter, easy to make operation.
* Data hold function to freeze the desired value on display.
* Microprocessor circuit ensures high accuracy and provides special functions and features.
* Operates from DC 1.5V (UM4/AAA) x 4 PCs batteries.
* Built-in low battery indicator.
* Durable, long-lasting components, enclosed in strong, compact ABS-plastic housing.

2. SPECIFICATIONS

Applications	Designed to check the moisture level of soil or other similar material.
Measuring Principal	Used the 2 pins electrode to measure the conductive ability of the species, then converter to the reading of % " Moisture content " of soil sample.
Display	LCD size : 28 mm x 19 mm.

Measurement Range	0 % to 50 % moisture content on soil.
Resolution	0.1 %.
Accuracy	± (5 % + 5 d) F.S. *@ 23 ℃ ± 5 ℃, F.S. : full scale.*
Circuit	Custom one-chip of microprocessor LSI circuit.
Probe	2 pins moisture electrode.
Data Hold	Freeze the display reading.
Sampling Time	Approx. 0.8 second.
Operating Temperature	0 to 50 ℃.
Operating Humidity	Less than 80% R.H.
Power Supply	DC 1.5 V battery (UM4/AAA) x 4 PCs,
Power Current	Approx. DC 5 mA
Weight	267 g/ 0.58 LB. *@ Battery is included.*
Dimension	*Meter body :* 172 x 40 x 40 mm (6.8" x 1.6" x 1.6").
	Probe body : 202 mm x Dia. 10 mm 8" x Dia. 0.4" .
	Total length (meter + probe) : 374 mm (14.7 ").
Accessories Included	Instruction manual..... 1 PC.

3. FRONT PANEL DESCRIPTION

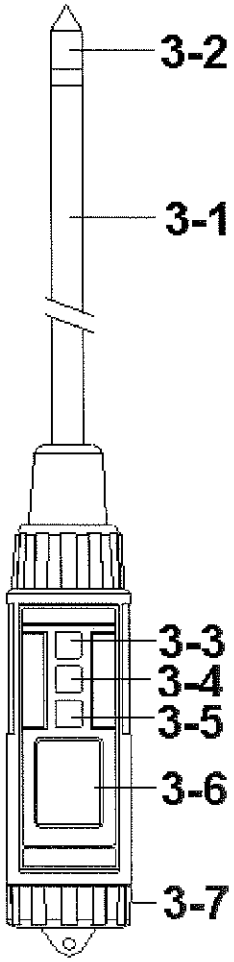

Fig. 1

3-1 Soil moisture Probe
3-2 Moisture Sensing Head
3-3 Power Button
3-4 Hold Button
3-5 REC Button
3-6 LCD Display
3-7 Battery Compartment/Cover

4. MEASURING PROCEDURE

4-1 Soil measurement

1) Turn on the meter by pressing the " Power Button " (3-3, Fig. 1) momentarily.

   ** Press the " Power Button " (3-3, Fig. 1) momentarily again will turn off the meter.*

2) Insert the " Moisture sensing head " (3-2, Fig. 1) into the measured soil.

   It is recommended that probe head should be inserted into the soil at least 10 cm when make the measurement..

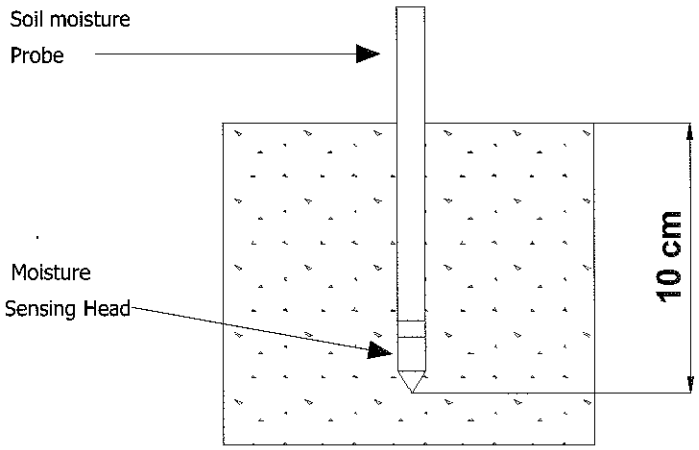

Fig. 2

Consideration :
If the sample soil under testing has a high moisture content it may take a few minutes to obtain a stable reading.

4-2 Data Hold

* During the measurement, press the " Hold Button " (3-4, Fig. 1) momentarily to hold the measured value. The LCD will show a " HOLD " symbol.
* Press the" Hold Button " once again to release the data hold function.

4-3 Data Record (Max., Min. reading)

1) The data record function records the maximum and minimum readings. Press the " REC Button " (3-5, Fig. 1) momentarily to start the Data Record function, shows " REC " on the display.
2) With the " REC " symbol on the display.
   a) Press the " REC Button " (3-5, Fig. 1) momentarily, the " REC MAX " symbol along with the maximum value will appear on the display.
   b) Press the " REC Button " (3-5, Fig. 1) momentarily again, the " REC MIN " symbol along with the minimum value will appear on the display.

   ** When display shows " REC MAX " or " REC MIN ", press the " Hold Button " (3-4, Fig. 1) momentarily will delete the max. (min.) value, the display will show the " REC. " only and execute the memory function continuously.*

   c) To exit the memory record function, press the " REC " button for 2 seconds at least. The display will revert to the current reading, not show " REC "

5. BATTERY REPLACEMENT

* Replace the batteries when the left corner of the LCD displays the low battery icon " ", using 4 fresh 1.5 V (UM4, AAA) batteries.
* To change the batteries, open (rotate clockwise direction) the " Battery Cover " (3-7, Fig. 1).
* Make sure the " Battery cover " (3-7, Fig 1) is secured after changing the batteries.

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Specialty Tools & Instruments™

GENERAL TOOLS & INSTRUMENTS™
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DSMM500 User's Manual

Specifications subject to change
without notice
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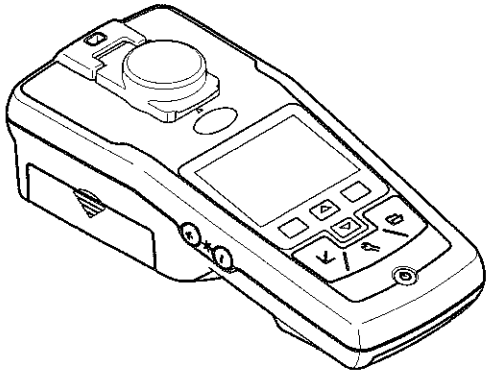
NOTICE - WE ARE NOT RESPONSIBLE FOR TYPOGRAPHICAL ERRORS.
MAN#DSMM500 04/09

DOC022.97.80041

2100Q and 2100Q*is*

04/2013, Edition 2



Basic User Manual
Manuel d'utilisation de base
Manual básico del usuario
Manual Básico do Usuário
基本用户手册
基本取扱説明書
기본 사용 설명서

English 3
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Specifications

Specifications are subject to change without notice.

Specification	Details
Measurement method	Ratio turbidimetric determination using a primary nephelometric light scatter signal (90°) to the transmitted light scatter signal.
Regulatory	**2100Q**: Meets EPA Method 180.1 **2100Q*is***: Meets ISO 7027
Lamp source	**2100Q**: Tungsten filament lamp **2100Q*is***: Light-emitting diode (LED) at 860 nm
Range	0–1000 NTU (FNU)
Accuracy	±2% of reading plus stray light from 0–1000 NTU (FNU)
Repeatability	±1% of reading or 0.01 NTU (FNU), whichever is greater
Resolution	0.01 NTU on lowest range
Stray light	≤ 0.02 NTU (FNU)
Signal averaging	Selectable on or off
Detector	Silicon Photodiode
Reading modes	Normal (Push to Read), Signal Averaging or Rapidly Settling Turbidity™
Calibration options	Single step RapidCal™ for Low-Level Regulatory Reporting from 0–40 NTU (FNU) Full range calibration from 0–1000 NTU (FNU) Calibration to degrees of turbidity
Calibration logger	Records the last 25 successful calibrations
Verification logger	Logs the last 250 successful verifications
Data logger	500 records

Specification	Details
Power requirement	AC 100–240 V , 50/60 Hz (with power or USB/power module) 4 AA alkaline batteries Rechargeable NiMH (for use with USB/power module)
Operating conditions	Temperature: 0 to 50 °C (32 to 122 °F) Relative Humidity: 0–90% at 30 °C, 0–80% at 40 °C, 0–70% at 50 °C, noncondensing
Storage conditions	–40 to 60 °C (–40 to 140 °F), instrument only
Interface	Optional USB
Sample required	15 mL (0.5 oz.)
Sample cells	Round cells 60 x 25 mm (2.36 x 1 in.) borosilicate glass with screw caps
Dimensions	22.9 x 10.7 x 7.7 cm (9.0 x 4.2 x 3.0 in.)
Weight	530 g (1.17 lb) without batteries 620 g (1.37 lb) with four AA alkaline batteries
Meter enclosure rating	IP67 (closed lid, battery and module compartment excluded)
Protection class	Power supply: Class II
Certification	CE certified
Warranty	1 year (EU: 2 years)

General information

In no event will the manufacturer be liable for direct, indirect, special, incidental or consequential damages resulting from any defect or omission in this manual. The manufacturer reserves the right to make changes in this manual and the products it describes at any time, without notice or obligation. Revised editions are found on the manufacturer's website.

Safety information

NOTICE

The manufacturer is not responsible for any damages due to misapplication or misuse of this product including, without limitation, direct, incidental and consequential damages, and disclaims such damages to the full extent permitted under applicable law. The user is solely responsible to identify critical application risks and install appropriate mechanisms to protect processes during a possible equipment malfunction.

Please read this entire manual before unpacking, setting up or operating this equipment. Pay attention to all danger and caution statements. Failure to do so could result in serious injury to the operator or damage to the equipment.

Make sure that the protection provided by this equipment is not impaired. Do not use or install this equipment in any manner other than that specified in this manual.

Use of hazard information

⚠ DANGER

Indicates a potentially or imminently hazardous situation which, if not avoided, will result in death or serious injury.

⚠ WARNING

Indicates a potentially or imminently hazardous situation which, if not avoided, could result in death or serious injury.

⚠ CAUTION

Indicates a potentially hazardous situation that may result in minor or moderate injury.

NOTICE

Indicates a situation which, if not avoided, may cause damage to the instrument. Information that requires special emphasis.

Precautionary labels

Read all labels and tags attached to the instrument. Personal injury or damage to the instrument could occur if not observed. A symbol on the instrument is referenced in the manual with a precautionary statement.

	This is the safety alert symbol. Obey all safety messages that follow this symbol to avoid potential injury. If on the instrument, refer to the instruction manual for operation or safety information.
	This symbol indicates that a risk of electrical shock and/or electrocution exists.
	Electrical equipment marked with this symbol may not be disposed of in European public disposal systems after 12 August of 2005. In conformity with European local and national regulations (EU Directive 2002/96/EC), European electrical equipment users must now return old or end-of-life equipment to the Producer for disposal at no charge to the user. ***Note:*** *For return for recycling, please contact the equipment producer or supplier for instructions on how to return end-of-life equipment, producer-supplied electrical accessories, and all auxiliary items for proper disposal.*

Certification

Canadian Radio Interference-Causing Equipment Regulation, IECS-003, Class A:

Supporting test records reside with the manufacturer.

This Class A digital apparatus meets all requirements of the Canadian Interference-Causing Equipment Regulations.

Cet appareil numérique de classe A répond à toutes les exigences de la réglementation canadienne sur les équipements provoquant des interférences.

FCC Part 15, Class "A" Limits

Supporting test records reside with the manufacturer. The device complies with Part 15 of the FCC Rules. Operation is subject to the following conditions:

1. The equipment may not cause harmful interference.

2. The equipment must accept any interference received, including interference that may cause undesired operation.

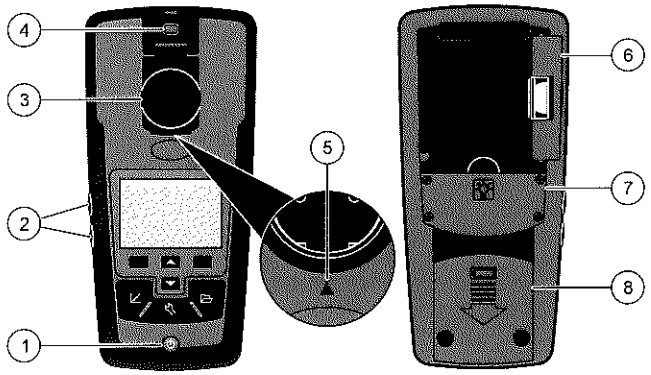
Changes or modifications to this equipment not expressly approved by the party responsible for compliance could void the user's authority to operate the equipment. This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to Part 15 of the FCC rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference, in which case the user will be required to correct the interference at their expense. The following techniques can be used to reduce interference problems:

1. Disconnect the equipment from its power source to verify that it is or is not the source of the interference.
2. If the equipment is connected to the same outlet as the device experiencing interference, connect the equipment to a different outlet.
3. Move the equipment away from the device receiving the interference.
4. Reposition the receiving antenna for the device receiving the interference.
5. Try combinations of the above.

Product overview

The 2100Q and 2100Q*is* portable turbidimeters measure turbidity from 0 to 1000 NTU (FNU). Primarily for field use, the portable meter operates on four AA batteries. Data can be stored and transferred to a printer, computer or USB storage device.

Figure 1 Product overview

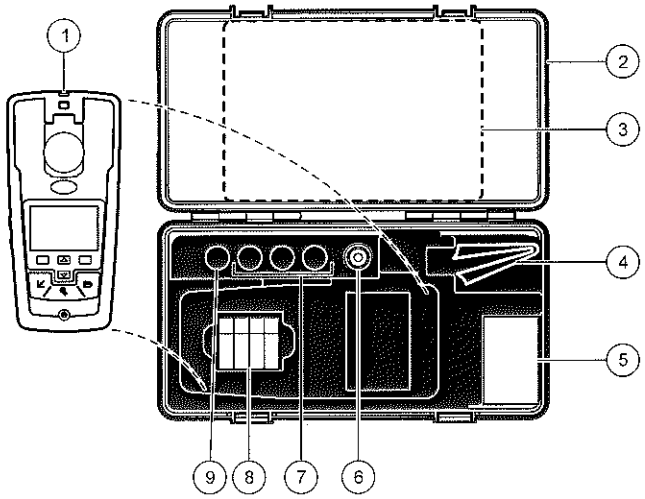


1 Power on or off	**5** Alignment arrow
2 Backlight keys (+ and -)	**6** Module
3 Sample cell holder with lid	**7** Lamp compartment
4 Attachment for lanyard	**8** Battery compartment

Product components

Refer to Figure 2 to make sure that all components have been received. If any of these items are missing or damaged, contact the manufacturer or a sales representative immediately.

Figure 2 2100Q and 2100Q*is* components



1 2100Q or 2100Q*is* turbidimeter	**6** Silicone oil
2 Carrying case	**7** 20, 100 and 800 NTU StablCal calibration standards
3 User manual and Quick reference guide	**8** AA alkaline batteries (pk/4)
4 Oiling cloth	**9** StablCal 10 NTU verification standard
5 1" sample cell (10 mL) with cap (pk/6)	

Installation

⚠CAUTION

Multiple hazards. Only qualified personnel must conduct the tasks described in this section of the document.

Install the battery

⚠WARNING

Explosion hazard. An expired battery can cause hydrogen gas buildup inside the instrument. Replace the battery before it expires. Do not store the instrument for long periods with a battery installed.

⚠WARNING

Potential fire hazard. Use only alkaline or nickel metal hydride batteries (NiMH) in the meter. Other battery types or incorrect installation can cause a fire. Never mix battery types in the meter.

NOTICE

The battery compartment is not waterproof. If the battery compartment becomes wet, remove and dry the batteries and dry the interior of the compartment. Check the battery contacts for corrosion and clean them if necessary.

NOTICE

When using nickel metal hydride (NiMH) batteries, the battery icon will not indicate a full charge after freshly charged batteries have been inserted (NiMH batteries are 1.2 V versus 1.5 V for alkaline batteries). Even though the icon does not indicate complete charge, 2300 mAH NiMH batteries will achieve 90% of instrument operation lifetime (before recharge) versus new alkaline batteries.

NOTICE

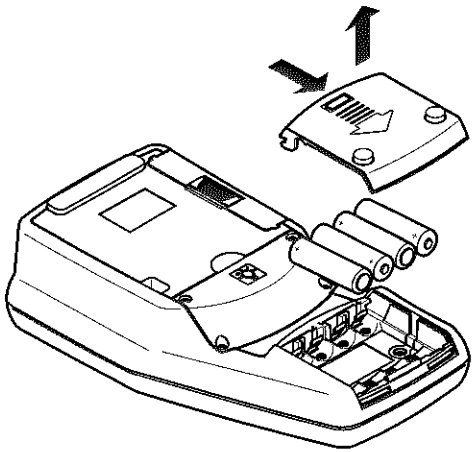
To avoid potential damage to the meter from battery leakage, remove the meter batteries prior to extended periods of non-use.

The meter can be powered with AA alkaline or rechargeable NiMH batteries. To conserve battery life, the meter will power off after 10 minutes of inactivity, the backlight powers off after 30 seconds. This time can be changed in the Power Management menu.
Note: *Rechargeable batteries will only be recharged with the USB/power module. Refer to the module documentation for further information.*

For battery installation refer to Figure 3.

1. Remove the battery cover.
2. Install 4 AA alkaline or 4 AA nickel metal hydride (NiMH) batteries. Make sure that the batteries are installed in the correct orientation.
3. Replace the battery cover.

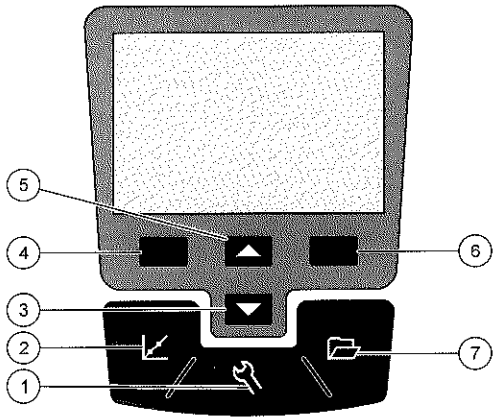
Figure 3 Battery installation



User interface and navigation

User interface

Figure 4 Keypad description

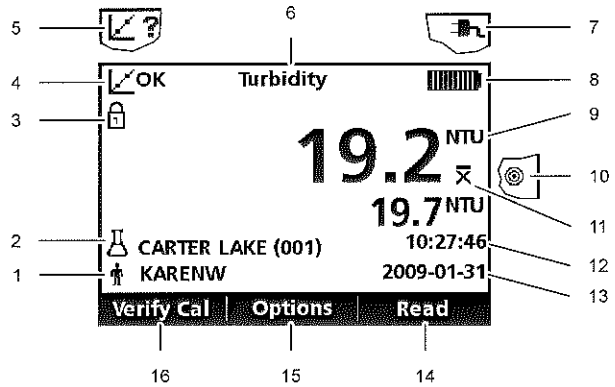


1 **SETTINGS** key: select menu options for setting up the meter	5 **UP** key: scroll through menus, enter numbers and letters
2 **CALIBRATION** key: shows calibration screen, start calibration, select cal options	6 **RIGHT** key (contextual): read turbidity sample, selects or confirms options, opens/jumps to sub-menus
3 **DOWN** key: scroll through menus, enter numbers and letters	7 **DATA MANAGEMENT** key: view, delete or transfer stored data
4 **LEFT** key (contextual): access for calibration verification, cancels or exits the current menu screen to the previous menu screen	

Display description

The measurement screen shows the turbidity, unit, calibration status, date and time, operator ID (if setup) and sample ID (if setup). Refer to Figure 5.

Figure 5 Single screen display



1 Operator identification	**9** NTU (Nephelometric Turbidity Unit) or FNU (Formazin Turbidity Unit)
2 Sample identification	**10** Reading mode: Rapidly Settling Turbidity (Target icon)
3 Stability or display lock indicator	**11** Reading mode: Signal Average (X-bar icon)
4 Calibration status indicator (Calibration OK=pass)	**12** Time
5 Calibration status indicator (Calibration ?=fail)	**13** Date
6 Parameter title	**14** Read (contextual: OK, Select)
7 AC power icon	**15** Options (contextual)
8 Battery icon	**16** Verification calibration

Navigation

The meter contains a Settings menu, Reading Options menu, Calibration Options menu and Calibration Verification Options menu to change various options. Use the **UP** and **DOWN** keys to highlight different options. Push the **RIGHT** key to select an option. There are two ways to change options:

1. Select an option from a list: Use the **UP** and **DOWN** keys to select an option. If check boxes are shown, more than one option can be selected. Push the **LEFT** key under Select.

   Note: *To deselect check boxes, push the **LEFT** key under Deselect.*
2. Enter an option value using the arrow keys:
   Push the **UP** and **DOWN** keys to enter or change a value.
3. Push the **RIGHT** key to advance to the next space.
4. Push the **RIGHT** key under **OK** to accept the value.

Startup

Turn the meter on and off

Push the **ON/OFF** key to turn on or turn off the meter. If the meter does not turn on, make sure that the batteries, or the module, are properly installed or that the AC power supply is properly connected to an electrical outlet.

Note: *The Auto-Shutoff option can also be used to turn off the meter. Additional information is available on the manufacturer's website.*

Change the language

There are three options to set the language:

- The display language is selected when the meter is powered on for the first time.
- The display language is selected when the power key is pushed and held.
- The language can be changed from the Settings menu.

1. Select a language from the list. Confirm with **OK**.
2. Push **Done** when the update is complete.

Change the date and time

The date and time can be changed from the Date & Time menu.

1. Push the **SETTINGS** key and select Date & Time.
2. Update the time and date information:

Option	Description
Format	Select one of the formats for the date and time: yyyy-mm-dd 24h yyyy-mm-dd 12h dd-mm-yyyy 24h dd-mm-yyyy 12h mm/dd/yyyy 24h mm/dd/yyyy 12h
Date	Enter the current date
Time	Enter the current time

The current date and time will be shown on the display.

After the date and time setup, the meter is ready to take a reading.

Standard operation

Use a sample ID

The sample ID tag is used to associate readings with a particular sample location. If assigned, stored data will include this ID.

1. Select **Sample ID** in the Settings menu.
2. Select, create or delete a sample ID:

Option	Description
Current ID	Select an ID from a list. The current ID will be associated with sample data until a different ID is selected.
Create a New Sample ID	Enter a name for a new sample ID.
Delete Sample ID	Delete an existing sample ID.

Use an operator ID

The operator ID tag associates readings with an individual operator. All stored data will include this ID.

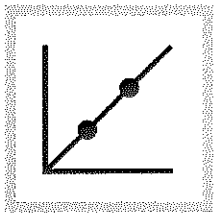
1. Select **Operator ID** in the Settings menu.
2. Select, create or delete an operator ID:

Option	Description
Current ID	Select an ID from a list. The current ID will be associated with sample data until a different ID is selected.
Create a New Operator ID	Enter a name for a new operator ID (maximum 10 names can be entered).
Delete Operator ID	Delete an existing operator ID.

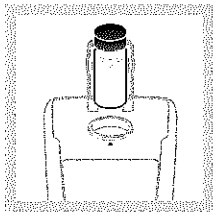
Advanced operation

Calibrate the turbidimeter with StablCal® Standards

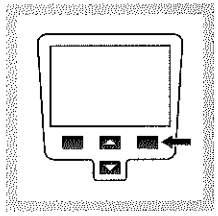
Note: *For best accuracy use the same sample cell or four matched sample cells for all readings during calibration. Insert the sample cell in the instrument cell compartment so the diamond or orientation mark aligns with the raised orientation mark in front of the cell compartment.*



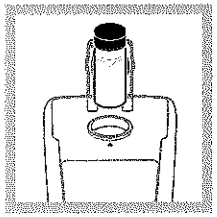
1. Push the **CALIBRATION** key to enter the Calibration mode. Follow the instructions on the display.
Note: *Gently invert each standard before inserting the standard.*



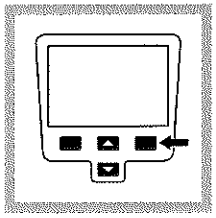
2. Insert the 20 NTU StablCal Standard and close the lid.
Note: *The standard to be inserted is bordered.*



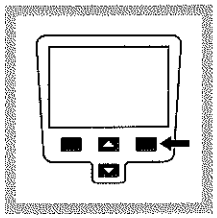
3. Push **Read**. The display shows Stabilizing and then shows the result.



4. Repeat Step 2 and 3 with the 100 NTU and 800 NTU StablCal Standard.
Note: *Push **Done** to complete a 2 point calibration.*



5. Push **Done** to review the calibration details.



6. Push **Store** to save the results.
After a calibration is complete, the meter automatically goes into the Verify Cal mode. Additional information is available on the manufacturer's website.

Reading modes

1. Push the **UP** or **DOWN** key to enter the Reading Options menu.
2. Select Reading Mode to select one of the following options:

Option	Description
Normal (Default setting)	The normal mode reads and averages three readings. The result is shown after the reading.

Option	Description
Signal Average	The Signal Average mode compensates for reading fluctuations caused by drifting of sample particles through the light path. The X-bar icon is shown on the display when signal averaging is on. The Signal Average mode measures 12 times and starts to show the average after three readings. The final result is the average of all 12 readings.
Rapidly Settling Turbidity™ (RST)	The Rapidly Settling Turbidity (RST) mode calculates and continuously updates the turbidity reading of the sample to a confidence of 95%, based on the accumulated trend of the real time measured values. The RST mode is best used on samples that settle rapidly and continuously change in value. The reading is based on a correctly prepared sample that is homogeneous at the beginning of the reading. It is best applied to samples that are greater than 20 NTU. The sample must be mixed thoroughly by inversion immediately before inserting it into the meter. The target icon is shown on the display when the Rapidly Settling Turbidity is on. The Rapidly Settling Turbidity reads and calculates five readings while showing intermediate results.

Maintenance

⚠CAUTION

Multiple hazards. Only qualified personnel must conduct the tasks described in this section of the document.

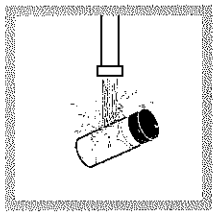
Clean the meter

The meter is designed to be maintenance-free and does not require regular cleaning for normal operation. Exterior surfaces of the meter may be cleaned as necessary.
Note: *Do not clean the meter with solvents to avoid damaging the material.*

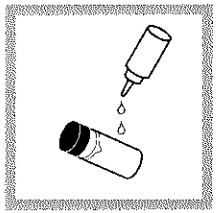
1. Clean the meter with a dust- and lint-free dry or slightly damp cloth. A mild soap solution can also be used for liposoluble contamination.

Apply silicone oil to a sample cell

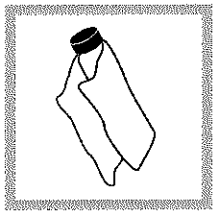
Sample cells and caps must be extremely clean and free from significant scratches. Apply a thin coating of silicone oil on the outside of the sample cells to mask minor imperfections and scratches that may contribute to light scattering.
Note: *Use only the provided silicone oil. This silicone oil has the same refractive index as the sample cell glass.*



1. Clean the inside and outside of the cells and caps by washing with a laboratory glass cleaning detergent. Follow with multiple rinses with distilled or demineralized water.



2. Apply a small bead of silicone oil from the top to the bottom of the cell.



3. Use the provided oiling cloth to spread the oil uniformly. Wipe off the excess so that only a thin coat of oil is left. Make sure that the sample cell is almost dry with little or no visible oil.
Note: *Store the oiling cloth in a plastic storage bag to keep the cloth clean.*

Store the sample cells

NOTICE

Do not air dry the sample cells.

Note: *Always store the sample cells with caps on to prevent the cells from drying.*

1. Fill the sample cells with distilled or demineralized water.
2. Cap and store the sample cells.
3. Wipe the outside of the sample cells dry with the a soft cloth.

Replace the battery

⚠WARNING

Explosion hazard. An expired battery can cause hydrogen gas buildup inside the instrument. Replace the battery before it expires. Do not store the instrument for long periods with a battery installed.

⚠WARNING

Potential fire hazard. Use only alkaline or nickel metal hydride batteries (NiMH) in the meter. Other battery types or incorrect installation can cause a fire. Never mix battery types in the meter.

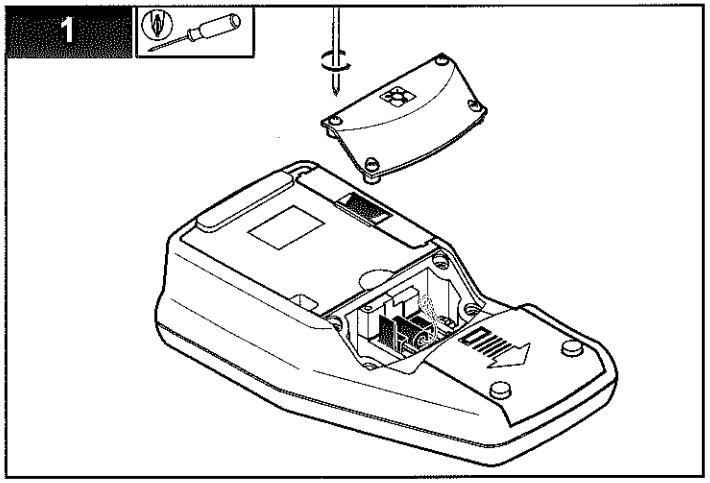
For battery replacement refer to Install the battery on page 6.

1. Remove the battery cover.
2. Remove the batteries.
3. Install 4 AA alkaline or 4 AA nickel metal hydride (NiMH) batteries. Make sure that the batteries are installed in the correct orientation.
4. Replace the battery cover.

Replace the lamp

⚠CAUTION

Burn Hazard. Wait until lamp cools down. Contact with the hot lamp can cause burns.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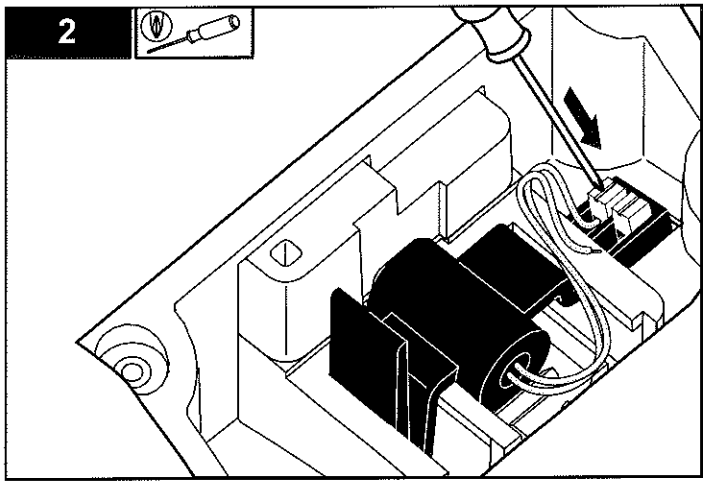
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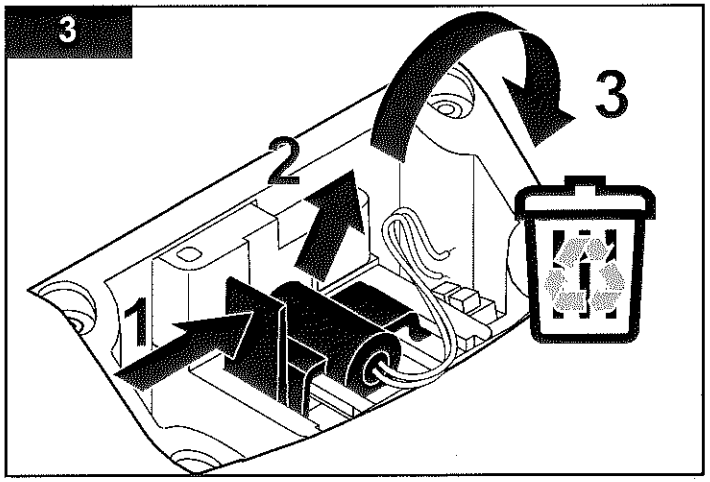
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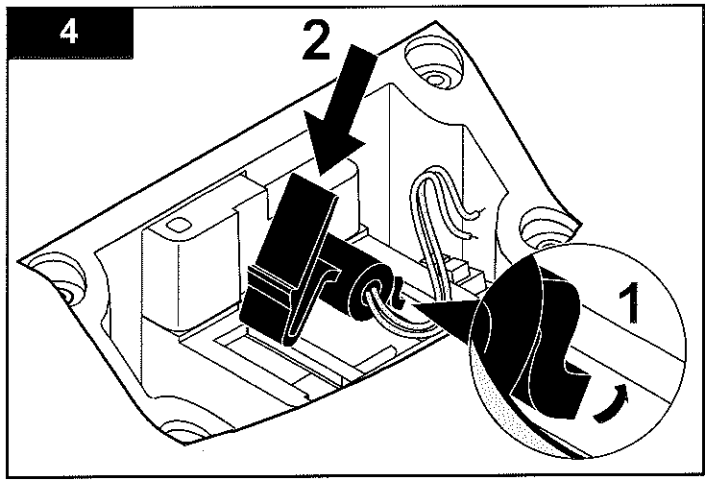
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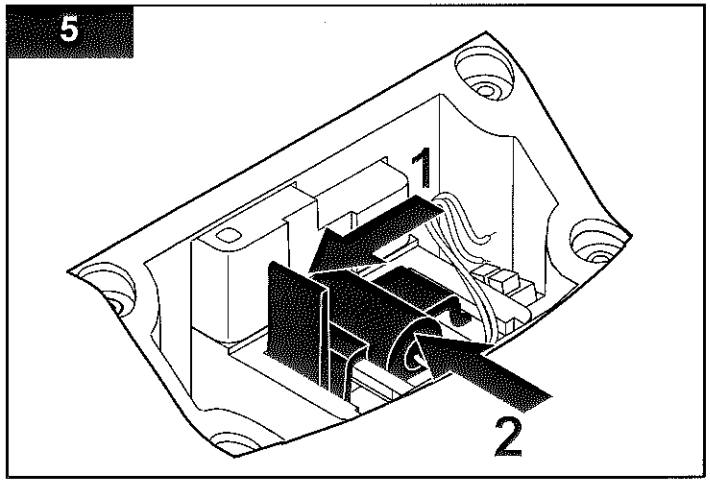
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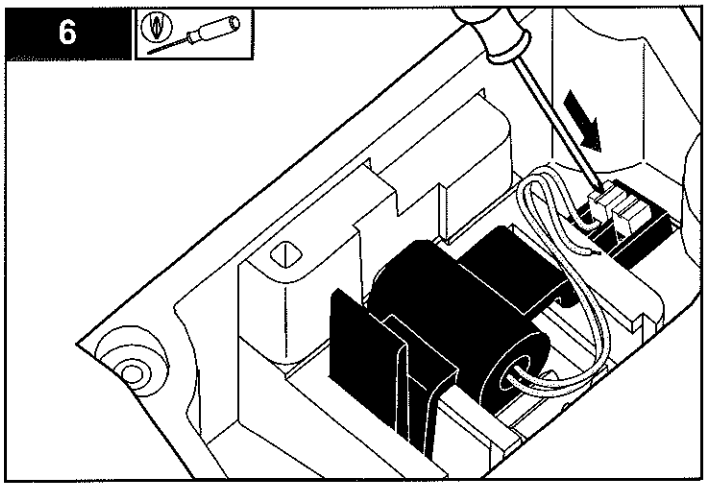
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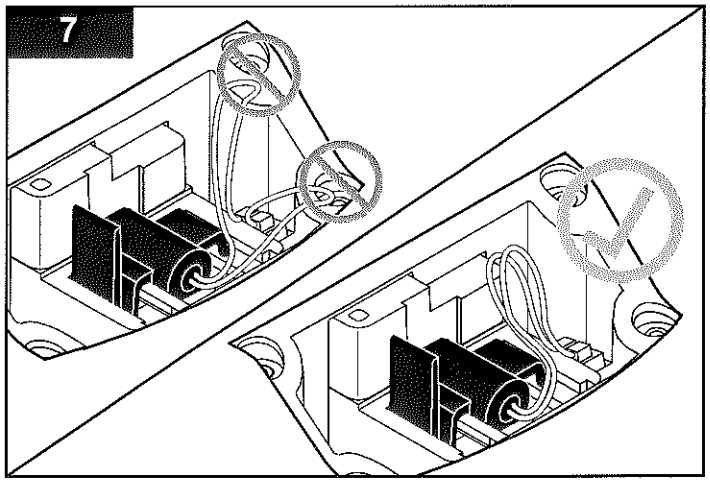
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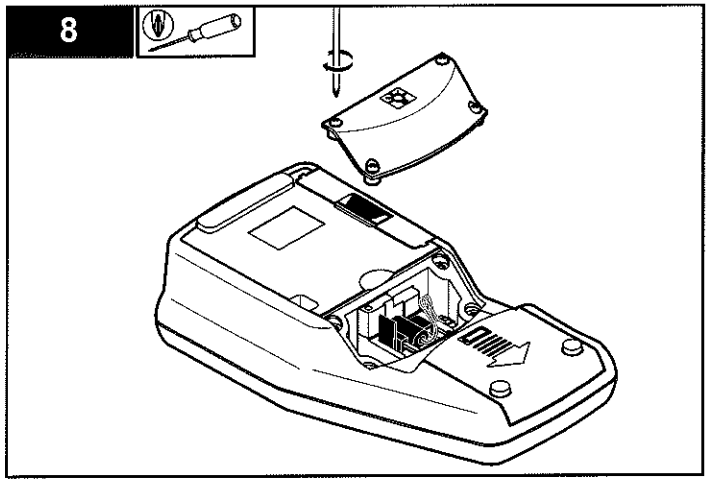

8

Troubleshooting

Refer to the following table for common problem messages or symptoms, possible causes and corrective actions.

Error/Warning	Description	Solution
Close lid and push Read.	The lid is open or lid detection failed.	Make sure that the lid is closed during reading and re-read.
Low Battery!	Battery is low.	• Insert new batteries • Connect USB/power module if rechargeable batteries are used
ADC Failure!	Hardware error causing reading to fail.	Repeat the reading.
Detector signal too low!	Insufficient light on the 180° detector.	• Check for obstructed light path. • Check the lamp.
Overrange!	Turbidity too high-caused probably by calibrating with RapidCal™ only.	• Calibrate the upper range. • Dilute the sample.
Underrange!	The measured absorbance is below the calibration range.	Repeat calibration
Please check the lamp!	Signals are too low on the 90° and 180° detector.	**2100Q:** The lamp is defective. Change the lamp (refer to Replace the lamp on page 12). **2100Q*is*:** Contact technical support.

Error/Warning	Description	Solution
Temperature too high! Switch off instrument.	Temperature has exceeded the meter limits (>60 °C or >140 °F).	Turn off the meter and let it cool down.
RST: Average value!	Solids are settling too slowly. The reading mode is not suitable for this sample.	Select Normal or Signal Average reading mode.
Confidence level is < 95%	The reading mode Rapidly Settling Turbidity did not meet the range of ≥ 95% confidence.	• Invert the sample several times so that the solids allocate. Repeat the reading again. • Switch to the Normal reading mode if the sample is stable and does not have settable solids.
Standard value out of range. Insert standard and push Read	Used incorrect standard value for the reading.	Insert the appropriate standard and read again.
ID already in use. Enter new ID	The Operator or Sample ID is unavailable as it is already assigned.	Create a new ID.
Error - Security Please set password before activating security	No password is created.	Create a new password.
Please enter at least one character.	Password must contain minimum of one character.	Create a password of at least one character.
Password incorrect. Please retry.	Incorrect password was entered.	Enter the appropriate password.

Error/Warning	Description	Solution
Please disconnect the USB cable from your computer.	Data storage does not respond while connected to the meter and the computer.	Disconnect the USB cable from the meter and try sending data again.
USB module memory full. Delete data and try again.	Data storage is full.	**1.** Connect USB/power module to the computer. **2.** Download the stored data to the computer. **3.** Delete Data Log on the module.
Delete Last Reading Failed!	Error in the data storage.	Turn the meter off and on. If the error message still occurs, contact technical support.
Delete Data Log failed!		
Can't read data set!		
Can't store data!		
Can't store to the Reading Log!		
Can't store to the Verify Cal Log!		
Error storing data!		
Error reading data!		

HORIBA

CODE: I1001578000

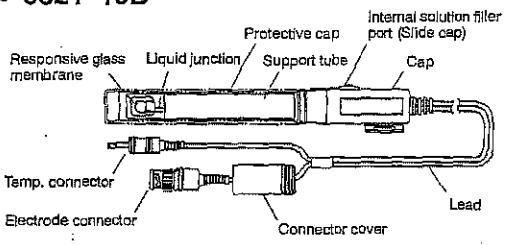
Instruction Manual for pH Electrodes

Thank you for purchasing a HORIBA pH electrode. Read this manual carefully, before using the electrode.

	Chemical solution	Glass fragments
⚠ Caution	The internal solution in the electrode uses very potassium chloride (3.33 mol/L-KCl). If the internal material comes in contact with the hands or skin, wash immediately with water. If the internal solution comes in contact with the eyes, flush with a large amount of running water, then seek medical advice.	Glass fragments can cause injury. The outer tube and tip of the electrode are made from glass. Be careful not to break them.

• Contents	· Electrode ···1 · Instruction manual ···1

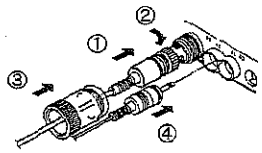
1 Specifications and parts description

Model/ Part name	Specification
• 9621-10D 	pH range: pH 0 to 14 Usable temp. range: 0°C to 100°C (At submergence measurement 0°C to 50°C) Strage temp. range: 0°C to 50°C Internal electrode: Silver/ silver chloride Reference electrode internal solution: 3.33 mol/L-KCl solution (#300) Liquid junction material: Porous ceramic Wetted material: Glass, silicon rubber, ceramic, polysulfone, polyacetal, polyimide, polyamide, PVC Electrode length: 150 mm (including a cap) Ext. diam. of wetted part: 16 mm Wetted part height: Approx. 15 mm (from the tip of electrode) Lead length: 1 m Maximum submergence depth: 1 m

Note▶▶▶ Body material is prastic. Do not use organic solvents (such as acetone or toluene)

2 Connecting to pH meter

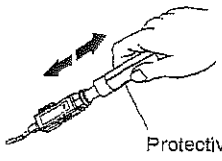
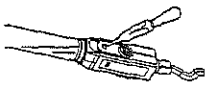
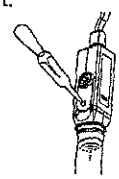
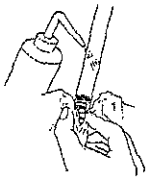
1. Insert the electrode connector into the connector port sleeve on the meter, after aligning with the pin. Do not insert the connector unless it is aligned properly with the connector port.
2. Press the electrode connector into the connector port on the meter, while turning the connector to the right.
3. Slide the connector cover over the connector. Then, push the cover in straight until it comes in light contact with the meter case. Do not turn the cover.
4. Insert the temperature connector into the jack on the meter. Insert the connector firmly, until the O-ring on the connector can no longer be seen.



3 Preparation

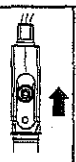
Note▶▶▶ White crystal (KCl) can adhere to Internal solution filler port. However, it it does not cause performance problem. Wash away the white crystal with pure water.

• For the first use or after a long-term interval

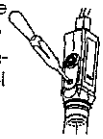
1. Remove the protective cap.	2. Remove the existing electrode internal solution with a dropper.	3. Fill the electrode with new internal solution (#300) until the solution level gets close to the filler port.	4. Wash the electrode tip well with pure water, then wipe it dry with filter paper.
			

4 Measurement (calibration)

1. Open the internal solution filler port. *1



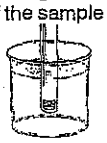
2. If necessary, fill the electrode with new internal solution until the solution level gets close to the filler port.



3. Wash the electrode with pure water and dry it with filter paper, prior to every immersion into standard/sample solution.

4. When there are air bubbles inside the responsive glass membrane, stir it lightly to remove bubbles.

5. Immerse the responsive glass membrane and liquid junction to the sample completely. Make sure that the surface of the internal solution inside the electrode is higher than the surface of the sample solution.



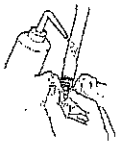
6. Stir the electrode lightly to remove bubbles.

*1 Be sure to keep the internal solution filler port open during measurement.
If it is clogged with the solution, accurate measurement cannot be performed. Wipe the solution with cloth.
*2 For calibration/ measurement on pH meter, refer to the instruction of the pH meter.

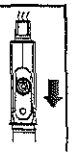
- **In submergence measurement**
  - Perform measurement with the internal solution filler port closed.
  - Pour the internal solution as much as possible, and close the internal solution filler port not to leave the bubbles inside the electrode.
  - Do not swing around the sensor, and do not pull the connection with the cable.
    In addition, do not hold the cable the electrode and pH meter hanging. It may cause breaking of wire.

5 Storage

1. Wash the electrode well with pure water to remove sample completely.

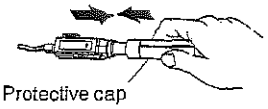


2. Close the slide cap.



3. Wash the inside of the protective cap with pure water, then add enough pure water to soak the sponge.

4. Attach the protective cap.



Protective cap

- Do not dry the responsive glass membrane and liquid junction.
  It may decline electrode responsiveness and sensitivity. If they are dry, immerse the tip of electrode in pure water for 12 hours or longer before use.
- Avoid storing the electrode in hot place or places with high humidity. Store the electrode indoors, out of direct sunlight.

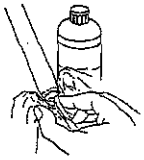
6 Maintenance

Note▶▶▶ Do not use organic solvents (such as acetone or tetrahydrofuran) or strong acid or alkaline cleansing solutions. Using such chemicals may damage the body of the electrode or cause a decline in performance.

- To maintain electrode performance, replace the internal solution approximately every two or three months. Refer to "3 Preparation."
- Dirt on the responsive glass membrane or liquid junction by the test solution may cause a decline in electrode response and sensitivity or measurement error. If the electrode is very dirty and cannot be washed clean using pure water, perform cleansing.

- Cleansing of electrode

1. Open the slide cap.
2. Immerse the responsive glass membrane and liquid junction in cleansing agent for approximately one hour until contamination is removed. The responsive glass membrane can also be cleaned by wiping it with cotton gauze containing cleansing agent.



3. Rinse the electrode with pure water completely.
4. Replace the internal solution.
   Refer to "3 Preparation."

Note▶▶▶ Be sure to perform calibration prior to measurement after cleansing the electrode.

Type of dirt	Cleansing agent
General dirt	Diluted neutral cleansing agent
Oil dirt	Alcohol, or diluted neutral cleansing agent
Inorganic dirt	Electrode cleaner (#220) or Hydrochloric acid approx. 1 mol/L
Protein dirt	Cleansing agent including protein-cutting enzyme
Liquid junction dirt	Electrode cleaner (#220)

7 For accurate measurement

For accurate measurement, refer to items below.

- Stir the sample with stirrer in measurement (calibration).
- Perform temperature compensation manually or automatically. (Refer to the instruction manual of pH meter.)
- Temperature of standard solution and sample should be the same.
- Immerse the responsive glass membrane and liquid junction to the sample completely. Make sure that the surface of the internal solution inside the electrode is higher than the surface of the sample solution.
- Rinse the electrode with the sample before measurement.
- Perform calibration before measurement.
  If measurement is performed everyday, perform calibration once or more a day.

8 Cautions on use

- Do not give the electrode strong shock.
- Do not use chapped or cracked electrode.
  Replace such electrode with a new one.
- Do not allow the connector to come in contact with water or dirty hands.
- Always use the #300 internal solution for reference electrode.
- If the responsive glass membrane or liquid junction is very dirty and cannot be washed clean using pure water, cleanse it depending on dirt condition. (Refer to "6 Maintenance.")
- Avoid continuous use of this electrode at temperatures higher than 60°C or at acidic or alkaline concentrations above 0.1 mol/L. Using the electrode under these conditions may harm performance and shorten the life of the electrode.
- Do not measure the solution which contains fluoride ion basically, which may corrodes the glass electrode.
- Do not suddenly immerse the electrode in samples where the temperature variation is 50°C or more, or the glass parts of the electrode may be damaged.
- Brown colored silver chloride can be detected inside the responsive glass membrane. However, it does not cause performance problem.
- Do not use the sensor in the place where usable temperature is out of specified range. Sensor may be damaged.
- White crystal can adhere to internal solution filler port or rubber stopper after storage of the electrode. However, it it does not cause performance problem.

For any question regarding this product, please contact your local agency, or inquire from the Customer Registration website (www.horiba.co.jp/register).

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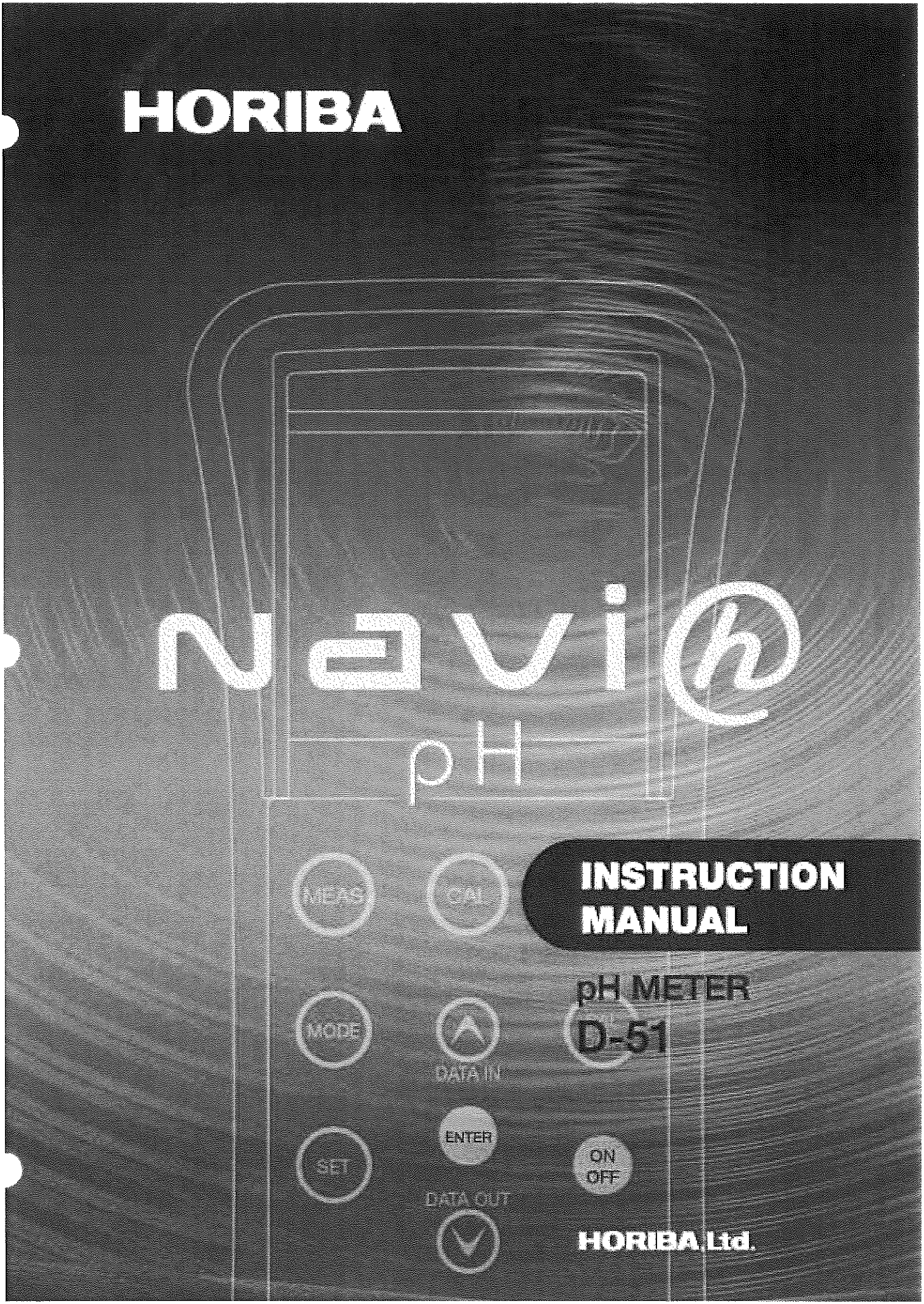
HORIBA



INSTRUCTION MANUAL

pH METER
D-51

HORIBA, Ltd.

Preface

Thank you for purchasing the D-51 pH meter .
This meter is designed with a compact body that can be held in one hand and features a water-resistant construction Note 1. It has a large-sized LCD display, which enables to use the varied functions by simple operations, and especially will be convenient to use on-location.
Carefully read this manual before using the meter.

Note 1: The water-resistant construction of this meter conforms to IP-67 of IEC 529, entitled "Water resistant testing and protection against penetration by solid matter for electrical machinery and equipment." To maintain the water-resistant construction of this meter, follow the instructions in this manual when using the meter.

IP-67 standards

- Dust does not get into internal parts.
- Water does not flow into internal parts when the meter is submerged 1 m below the surface of the water for 30 minutes, at a temperature differential between the water and the device of 5 ℃ or less.

HORIBA's Warranty and Responsibility

Your meter is covered by HORIBA's warranty for a period of one (1) year, under normal use. Although unlikely, if any trouble attributable to HORIBA should occur during this period, necessary exchange or repairs shall be conducted by HORIBA, free of charge. The warranty does not cover the following:

- Any trouble or damage attributable to actions or conditions specifically mentioned to be avoided in the operation manuals
- Any trouble or damage attributable to use of the meter in ways or for purposes other than those described in the operation manuals
- If any repairs renovations, disassembly, etc. are performed on this meter by any party other than HORIBA or a party authorized by HORIBA
- Any alteration to the external appearance of this pH meter attributable to scratches, dirt, etc. occurring through normal use
- Wear and tear to parts, the exchange of accessories, or the use of any parts not specified by HORIBA

HORIBA also shall not be liable for any damages resulting from any malfunctions of this product, any erasure of data, or any other uses of this product.

Unauthorized reprinting or copying of this operation manual

No unauthorized reprinting or copying of all or part of this operation manual is allowed. The utmost care has been used in the preparation of this operation manual. If, however, you have any questions or notice any errors, please contact the HORIBA customer service center printed on the back cover of this operation manual.

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■ CE Marking

This product is in conformity with the following directives and standards:

Directives:The EMC Directives 89/336/EEC
The Electrical Product Safety Directive 73/23/EEC

Standards: EN61326: 1997+A1:1998
(EMISSION: Class B, IMMUNITY Category: Minimum Requirement)
EN61010-1: 2001

Installation Environment

This product is designed for the following environment.
- Pollution degree 2
- Measurement category Ⅰ

WARNING:Do Not use the equipment for measurements within measurement categories
Ⅱ, Ⅲ and Ⅳ.

■ FCC Warning

This equipment has been tested and found to comply withthe limits for a Class A digital device, pursuant to part 15 of the FCC Rules.
These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment.
This equipment generates, uses, and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications.
Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be
required to correct the interference at his own expense.

■ Type and Definition of Signal Words

For the safety use, the meter is equipped with the Warning Labels to alert every operator and user to the possible risk and danger. Before using understanding each message.
The meaning of signal words are as follows:

(WARNING) This indicates an potentially hazardous situation which, if not avoided, will result in death or serious injury.

(CAUTION) This indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury. It may also be used to alert unsafe practices.

■ Safety Precautions

For the safety use, be sure to read the following precautions:

⚠ WARNING:

- Do not use any unspecified AC adapters.
  Heat or fire may occur to cause fire or accidents.
- Do not disassemble or modify the meter.
  Heat or fire may occur to cause fire or accidents.

⚠ CAUTION:

- Do not use the serial communication or AC adapter in the place that may possibly contact with moisture.
  It may cause fire, electric shock, or breakage.
- Part of the electrode is made of glass; handle with care not to break it.

● Indication

⚠ WARNING

This indicates an potentially hazardous situation which, if not avoided, will result in death or serious injury.

⚠ CAUTION

This indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury. It may also be used to alert unsafe practices.

— Note ———

This mark indicates the operation requires a special care and attention.

— Ref. ———

This mark indicates to which the reader should go for reference.

— HINT! ———

This mark indicates reference information.

■ Cautionary Items

● Precautions

- Do not give physical shock to the meter like dropping or hitting.
- Do not immerse the meter into alcohol, organic solvent, strong acid, strong alkaline, and other similar solutions. The meter contains ABS resin, acrylic resin, and various rubber products in its body.
- Do not use a hair-dryer for drying the meter. When the meter is dropped into water or get wet, wipe it using soft cloth.
- Perform the key operation by the fingers, not by the hard object like metal stick or rod.
- Be careful not to let water into the meter when the electrode connector is empty or the AC adapter or serial communications cable has been connected. In those states, the meter is not water-proof.
- To disconnect the electrode cable or interface cable, pull them out with holding the connector part. Do not pull the cable part; it may cause a breakage.
- Do not remove the battery gasket or twist it.
- When opening the battery case, make sure that no foreign matter is attached to the battery gasket.
- Do not use any unspecified batteries ; it may cause a breakage.

● Location of use and storage

- The place which room temperature is at 0 ℃ to 45 ℃
- The place which relative humidity is under 80% and free from condensation

Do not use or store the meter at;

- The place of much dust
- The place with strong vibration
- The place with direct sunlight
- The place with corrosive gas generation
- The place near from an air-conditioner
- The place with direct wind

● Move and Transportation of the meter

To transport the meter, use the packaging box at the delivery. Transportation by any unspecified packing methods may cause a breakage.

● Disposal

Standard solution used for the calibration must be under neutralization before the disposal. As for the disposal of the meter, treat it as an industrial waste.

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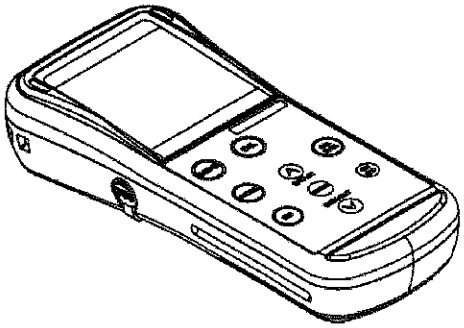
1 Overview of the Meter

This chapter explains the part names, how to connect the electrodes, how to replace the batteries, and precautions when using the meter.

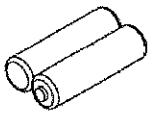
1.1 Package contents

The following items are shipped with each HORIBA pH meter package.

Meter (main unit) 1 unit



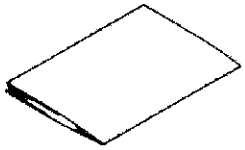
Dry-cell batteries 2 pcs.



Strap 1 pc

Soft case 1 pc

Operation manual 1 book



Note

To take measurements, you will need electrode(s).
Refer to "5.5 Spare and optional parts" page 79 when purchasing the electrode(s).

1.2 Functions

The D-51 features the following functions.

● Measurement items

Items	Required electrode/ standard solution
pH	pH electrode, pH standard solution
Temperature	—

● Functions

An overview of the functions found on HORIBA the D-51 is shown below.

Function	Explanation	Page No.
Data memory	Stores the data of measured values and temperature to the memory (max. 300 items)	page 29
pH repeatability check	Displays the difference between the calibration value and measured value after calibration.	page 26
pH calibration history display	Displays the date of calibration and the asymmetrical potential and sensitivity.	page 32
Clock	Displays the time on the screen and recorded data.	page 29
Auto Power OFF	Automatically turns off power if no operation is made after 30 minutes.	page 47
AC adapter connection	The meter can be AC-powered.	page 13

● Setting Items

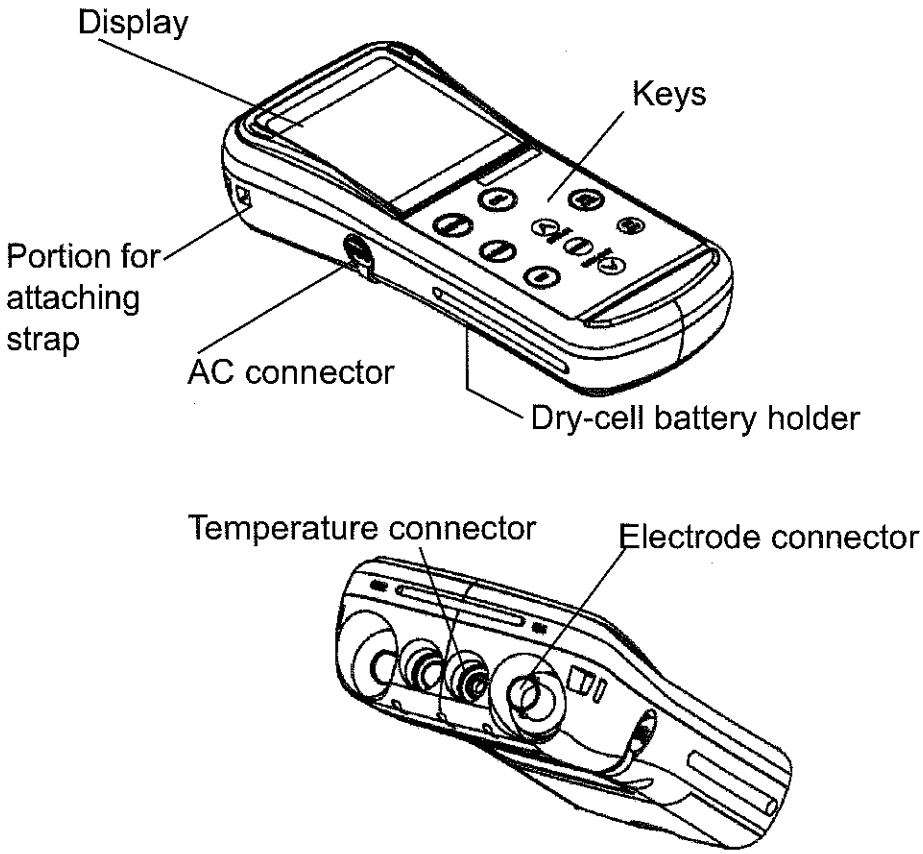
Function	Explanation	Page No.
pH standard solution setting	Enables standard solution used for calibration to be changed to NIST and US specifications settings.	page 37
Temperature compensation	Toggles between Automatic Temperature Compensation (ATC) mode that measures the sample temperature using the temperature sensor built-in the electrode and Manual Temperature Compensation (MTC) mode that uses user-specified temperature.	page 41

● Functions in Maintenance mode

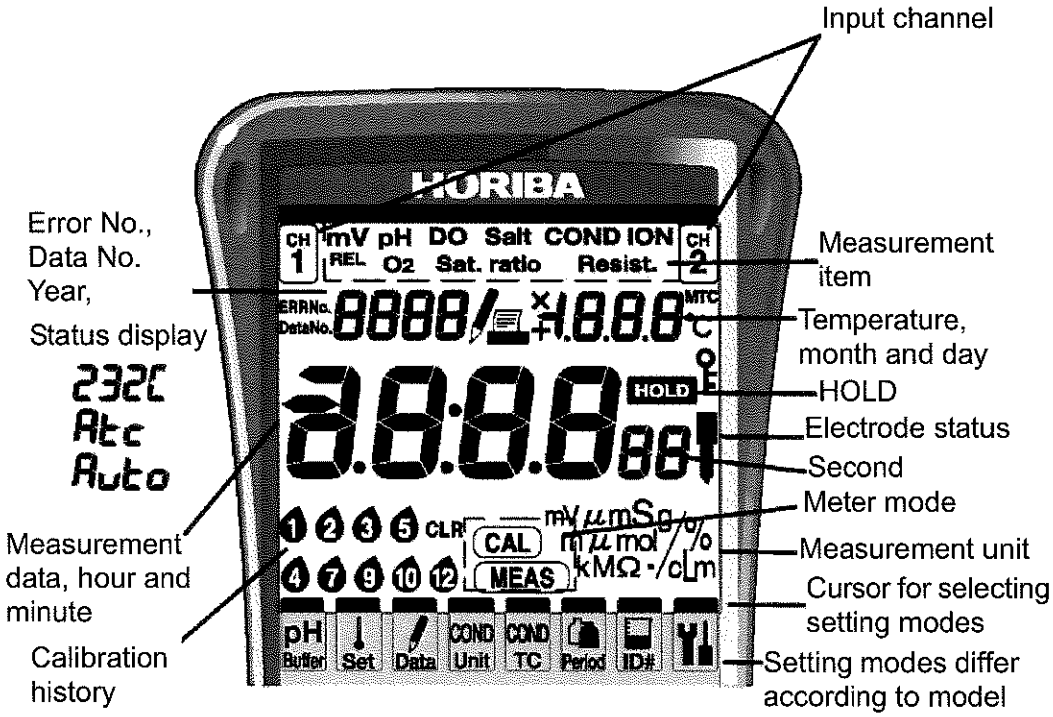
Function	Explanation	Page No.
LCD check	Enables check for whether or not all LCD segments are displayed.	page 44
Battery voltage check	Enables simple check of battery voltage.	page 45
Temperature display calibration	Adjusts the display of the temperature sensor to the actual temperature.	page 46
Auto Power OFF	Sets the function that automatically turns the power OFF if no keys are touched 30 minutes.	page 47
Remaining data memory	Displays the remaining memory.	page 48
Data memory clear	Deletes data in memory.	page 48
Initializing settings	Initializes all settings to the default values.	page 49

1.3 Part names

The D-51, pH meter has the following parts:



1.4 Explanation of display



Part name	Display	Contents
Input channel Measurement item	CH 1	Input channel 1
	pH	Displayed when measuring pH
Error No.	ERRNo.	Displayed when an error is generated
Data No.	DataNo.	Displayed when the data number has been set.
Status display — — —	001	Shows error number and data number.
	Atc	Displayed when temperature compensation function or automatic temperature compensation has been set.

Part name	Display	Contents
		Displayed during data memory function (for 3 sec.). Displayed while data in memory is being called up and when manual data memory is being called up, or blinks when automatic data memory is being called up.
	MTC	Displayed during manual temperature compensation. Not displayed during automatic temperature compensation.
HOLD	HOLD	Displayed while the data is held (HOLD status). Blinks during measurement or calibration.
Electrode status		(Only in pH Measurement mode) Not displayed: Normal Blinking: Cleaning is needed. Constant display: Replacement time is approaching.
Calibration history	❶ ~ ⓬	Calibration history display: Displayed after calibration for pH and ION electrodes as calibration history.
	CLR	When no calibration data is available: Displayed when no calibration has been performed for pH and ION electrodes.
Meter mode	MEAS	Displayed when in Measurement mode.
	CAL	Displayed when in Calibration mode.

1.5 Operation keys

This section describes the functions of the keys.

	Name	Description
MEAS	MEAS key	Returns to the Measurement mode. Starts measurement.
MODE	MODE key	Selects measurement item.
SET	SET key	Selects setting item.
CAL	CAL key	Enters the Calibration mode. Starts calibration.
^	UP key	Executes the data memory function. Increases numerical value.
	ENTER key	Establishes the setting.
V	DOWN key	Calls up data memory. Decreases numerical value.
CAL DATA	CAL DATA key	Calls up calibration data.
	ON/OFF key	Turns ON/OFF the power. This key takes effect only after pressed for one second to prevent accidental operation.

Note

The automatic power-off function is a default setting for this meter. The power is automatically turned OFF if no operation is performed after a period of approximately 30 minutes.

1.6 Connecting the electrodes

Connect the electrodes to the meter using the following procedure:

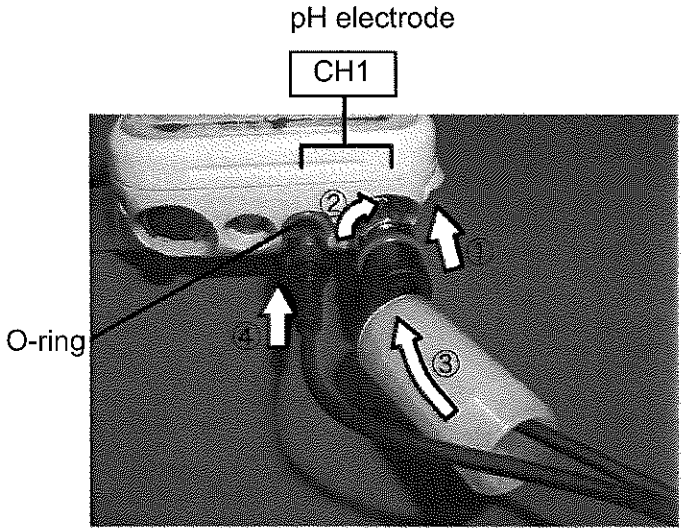
Note

- Do not allow any water to come into contact with the connector.
- Do not touch the connector with uncleaned hands.
- Hold the metal portion when turning the electrode conenctor.

The following connectors are used depending on electrode type:

CH1	Electrode connector:	pH electrode
	Temperature connector:	Temperature electrode for CH1

● Electrode connector (G-R electrode)



1. Insert the electrode connector, making sure to align the connector grooves with the pins in the connector port on the main unit (see photo, ①). Do not push the electrode with undue force when the pins are not properly aligned.

2. Push the electrode connector into the connector port while turning it clockwise, following the grooves (see photo, ① and ②).

3. Push the connector cover over the connector (see photo, ③), being careful to push it straight on without turning it.

Note

The meter will be waterproof only if this cover is placed properly over the connector.

● Temperature connector

1. Insert the temperature connector into the jack on the main unit until the O-ring on the electrode cannot be seen at all (see photo, ④).

Note

The meter will not be waterproof if the electrode is not inserted properly.

Note

When the temperature electrode is not connected (or is connected improperly), the automatic temperature compensation (ATC) will be 25°C.

1.7 Inserting/replacing the dry-cell batteries

The dry-cell batteries are not placed in the meter before shipping. To insert the batteries, follow the procedure below.
Note that if "ERR 2" appears on the display while using the meter, it indicates that the charge of the dry-cell batteries is running low. When this occurs, replace the batteries promptly.
Dry-cell battery type: AA alkaline

Note

- Insert the batteries, paying attention to the orientation of the battery poles ("+" and "- ").
- Removing the batteries will erase the clock data. To save the clock data, remove and replace the batteries while the meter is connected to the AC adapter (sold separately).
- Replace the batteries only after turning the power OFF. Any saved data will not be lost.
- When opening and closing the battery cover, be careful that no water gets inside the meter.
- Check that the rubber packing is not twisted and no foreign matter is stuck to it. Otherwise the meter may no longer be waterproof.

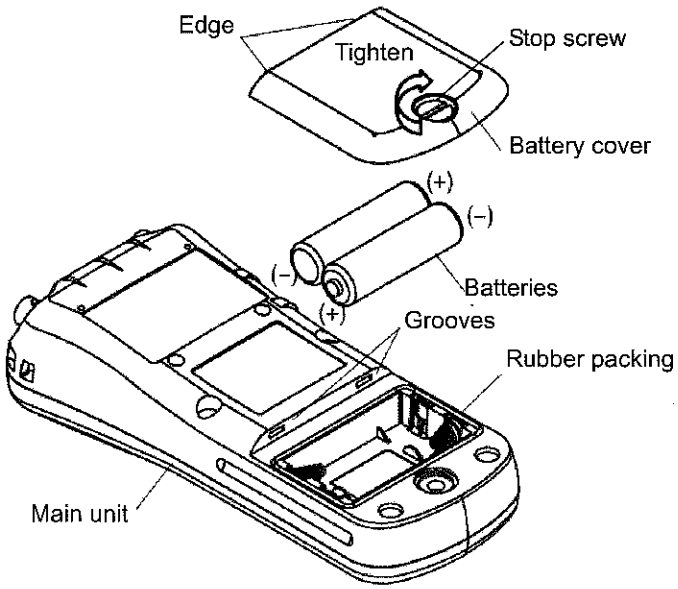
Note

The life of the batteries included with the meter may be short because the batteries were used for the operation check before shipping.

To insert/replace the batteries

1. Loosen the screw of the battery cover by using a coin or screwdriver, etc. The cover is constructed so that the stop screw cannot be completely removed and lost.
2. Pull up the screw, and remove the battery cover by sliding it out.

3. If there are old batteries inside, remove them.
4. Place the new batteries in the meter, verifying the orientation of the poles ("+" and "-").
5. Check that the rubber packing is not twisted and no foreign matter is stuck to it.
6. Insert the edge of the battery cover into the grooves on the meter, and then tighten the stop screw.



Note

Check that the rubber packing is twisted and no foreign matter is stuck to it. Otherwise the meter may no longer be waterproof.

Battery life

The table below shows the battery life of alkaline batteries during continuous use. The life of manganese batteries is about a half of the alkaline batteries.

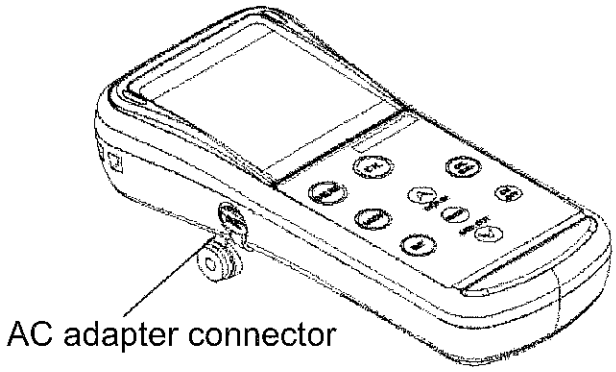
Battery life	approx. 200 hours

1.8 Connecting the AC adapter

When using the meter with an AC power supply, use the designated AC adapter (option).

AC adapter specifications

Supply voltage range	100 - 200 V AC
Frequency range	50/60 Hz
Current rating	Max 370 mA
Class2 Power supply	
Equipment protected by double insulation	
Indoor use only	
Supply voltage fluctuations allowed up to ± 10%	



Note

When the AC adapter is connected, the meter is no longer waterproof.
Be careful not to let water get into the meter.

2 Taking Measurements

This chapter explains how to take basic measurements.

2.1 Turning the meter ON/OFF

Pressing the ON/OFF key turns the power on/off. The ON/OFF key functions when it is pressed continuously for about one second to protect against accidental operation.

2.2 Settings required before measurement

The built-in clock allows you to record the date of calibration and data memory storage. When using the meter for the first time, be sure to set this clock.

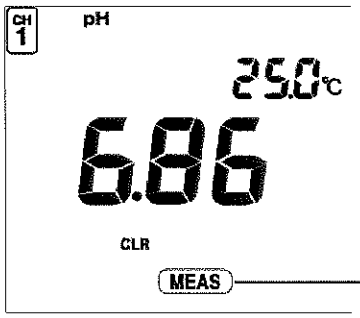
Ref.
"3.3 Displaying and setting the clock" page 34

2.3 Measurement modes

The D-51, pH meter has an Instantaneous Value Measurement mode and an Auto Hold Measurement mode for all components of the solution being measured.

● Instantaneous Value Measurement mode

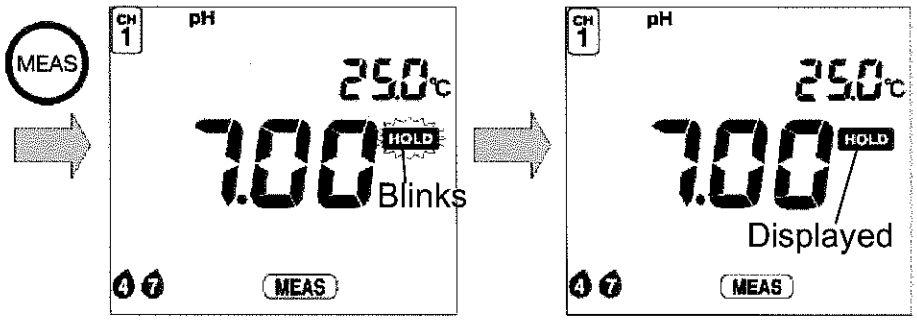
The D-51, pH meter performs instantaneous value measurement as the default measurement mode when the power is first turned ON and when the auto hold measurement is cancelled or cleared.
For this reason, the screen displayed when the meter is in the Instantaneous Value Measurement mode is called the "initial screen" in this manual.



Displayed

● Auto Hold Measurement mode

Auto Hold Measurement mode maintains the display of the value measured when the meter automatically judges that the measured value has stabilized. Press the MEAS key with the initial screeen displayed to make "HOLD" blink on the display. When the measured value becomes stable, "HOLD" will stop blinking and remain displayed, and the measured value will remain displayed. To clear the hold status or "stabilized" value (when "HOLD" is blinking), press the MEAS key.

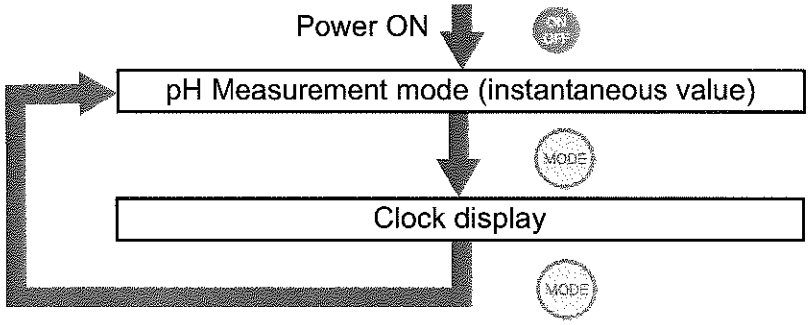


Criteria for judging stability

pH, ORP, ION measurement	: Within ±1 mV variance in potential after 10 seconds
Temperature measurement	: Within ±2°C variance after 10 seconds

2.4 Selecting the measurement modes

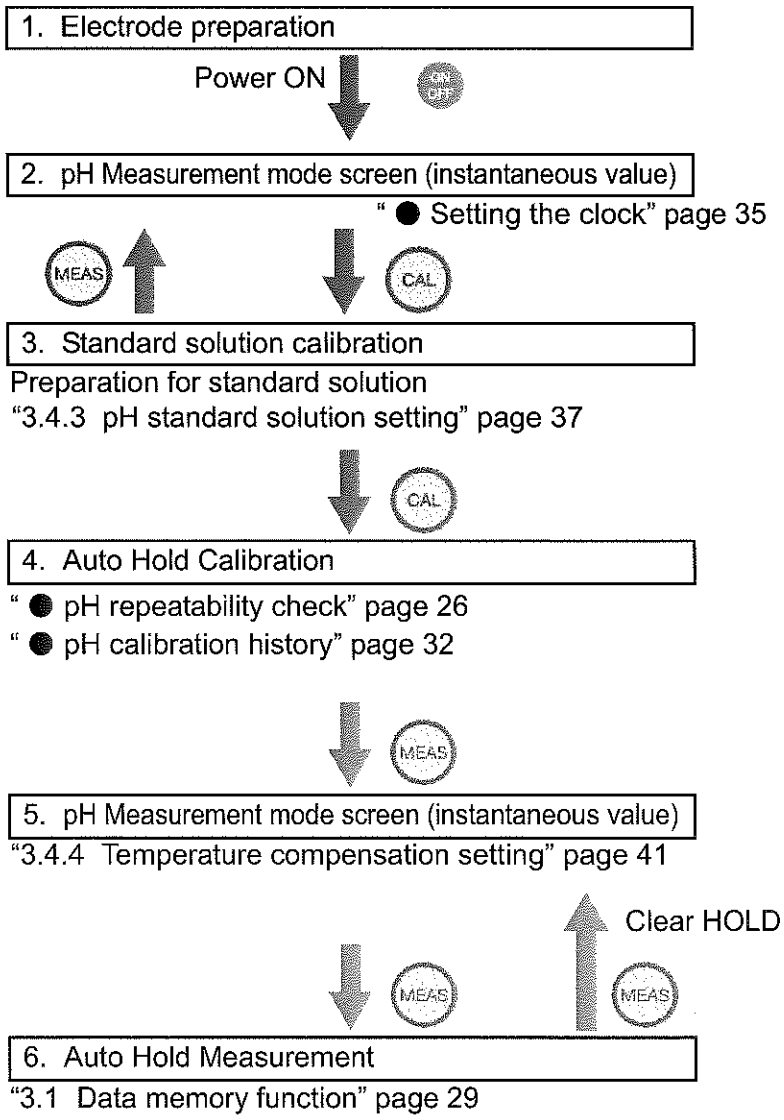
Pressing the MODE key changes the measurement mode. The last measurement mode item is the clock display. Pressing the MODE key once more returns the display to the first measurement mode.



2.5 Measuring pH

The following shows the operational flow for pH measurement.

● Measuring pH: basic operational flow



● Electrode preparation

Refer to the electrode instruction manual and make sure you have the necessary electrode(s).
Plastic-body pH electrode: 9621-10D
Glass-body pH electrode: 9611-10D
pH (micro) electrode: 9669-10D
pH (sleeve) electrode: 9677-10D

Chemical solution

Caution

The liquid inside the electrode is highly concentrated potassium chloride (3.33 mol/L KCl). If the internal solution in the electrode comes in contact with your hands or skin, wash immediately with water. If the internal solution comes in contact with your eyes, flush immediately with large amounts of water and seek treatment by a physician.

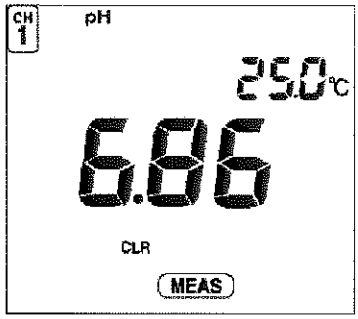
Glass fragments

Caution

Glass fragments can cause injury.
The outer tube of the electrode and the tip of the electrode are made of glass.
Use care not to break them.

● Entering the pH Measurement mode

1. Press the ON/OFF key.
   The initial screen will appear.



● Standard solution calibration

Perform a one-point calibration for making simple pH measurements; for more accurate measurements, perform at least a two-point calibration.

Note

Up to three points can be used for calibration. If you perform calibration for a fourth points, "ERR06 Calibration point error" is displayed.

Standard solutions for calibration are defaulted to pH 2, pH 4, pH 7, pH 9, and pH 12.

Ref.

"3.4.3 pH standard solution setting" page 37

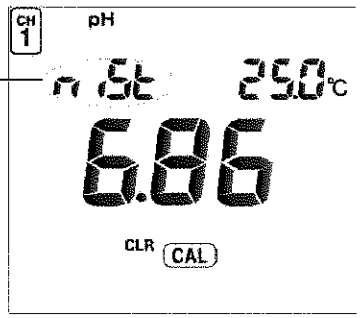
This section will explain how to conduct a two-point calibration using pH 7 and pH 4 standard solutions.

Calibration procedure

1. Press the CAL key while in the pH Measurement mode.

The meter enters the Calibration mode and >CAL< is displayed.

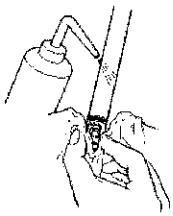
Displayed item differs depending on the standard solution setting.



Note

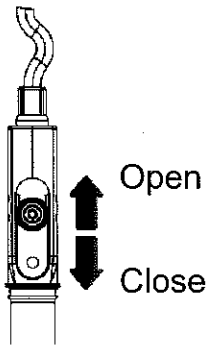
The mode cannot be changed during Auto Hold calibration (while "HOLD" is blinking or continually displayed.

2. Wash the tip of the electrode well with pure (de-ionized) water, and then wipe with filter paper or tissue paper.



3. Open the internal solution filler port.

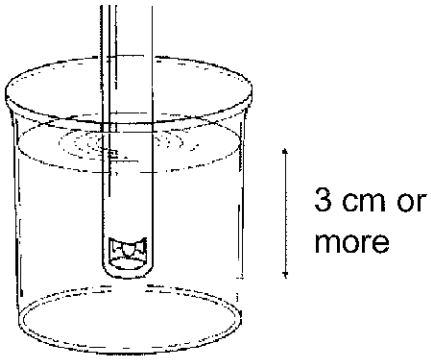
Leave the port open while calibration is taking place.



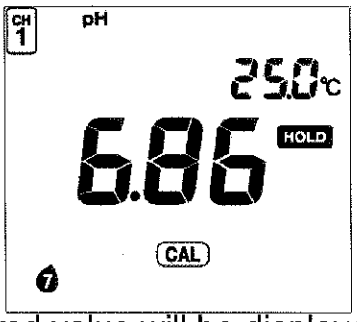
Calibration at first point

1. Immerse the tip of the electrode in a beaker containing pH 7 standard solution.

Immerse the pH electrode in the sample at least three centimeters.



2. Press the CAL key to start calibration.



The measured value will be displayed, and "HOLD" will blink until the reading stabilizes.

When the value stabilizes, "HOLD" will stop blinking and the calibrated value will be displayed.

The 7 bottle mark will be displayed, indicating that calibration was conducted with pH7 standard solution

Note

To stop the calibration:
Press the CAL key while the HOLD mark is blinking.
To establish the calibration:
Press the ENTER key while the HOLD mark is blinking.
To redo the calibration:
Press the CAL key after the HOLD mark is displayed.

Calibration at second point

1. Wash the electrode well again with pure (de-ionized) water, and then wipe with filter paper or tissue paper.

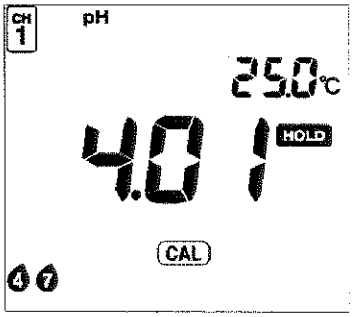
2. Immerse the tip of the electrode in a beaker containing pH 4 standard solution.

3. Press the CAL key to start calibration.

   The measured value will be displayed, and "HOLD" will blink until the reading stabilizes.

   When the value stabilizes, "HOLD" will stop blinking and the calibrated value will be displayed.

   The ❹ bottle mark will be displayed, indicating that calibration was conducted with pH 4 standard solution.



4. Press the MEAS key to return to the pH Measurement screen.

Note

While calibrations are being performed in the Calibration mode, redoing a calibration only updates the calibration data for the pertinent standard solution. If a calibration is redone after the meter is returned to the Measurement mode, however, the calibration is conducted on the initial status of the meter; i.e., all the previous calibration data is cleared.

Note

The example of calibration at second point has explained the calibration process using the order from pH 7 to pH 4. However, the calibration order of the standard solutions can be arbitrarily chosen.

Electrode status

You can check the status of the electrode after calibration.

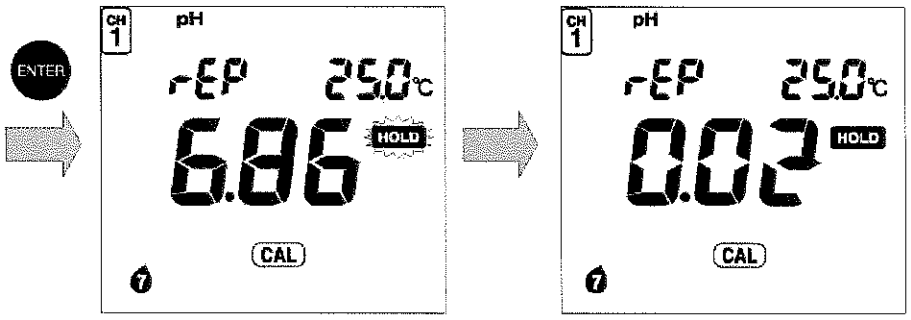
Item	Description
,ERR Not displayed	The electrode is in good condition. Electrode sensitivity is from 93% to 100%.
Blinking	Electrode sensitivity has dropped to the level of 90% to 93%. ・Make sure that you are using the right standard solution. ・Clean the electrode. " ● Washing the electrodes" page 54
Displayed	Electrode sensitivity has dropped to the level of 85% to 90%. "● ERR No.05 Electrode sensitivity error (pH)" page 59
ERR No.04	Asymmetrical potential error " ● ERR No.04 Asymmetric potential error" page 58
ERR No.05	Sensitivity error "● ERR No.05 Electrode sensitivity error (pH)" page 59

● pH repeatability check

The repeatability of the calibration can be checked if the calibration has been performed with pH 7 standard solution of NIST or US.
The repeatability check is operable only once after calibration.

1. After calibration and while still in the Calibration mode, immerse the electrode in pH 7 standard solution and press the ENTER key.

   The difference between the calibrated value and measured value is displayed.



Note

There is no problem in measurement accuracy if the difference is within ± 0.05 pH.

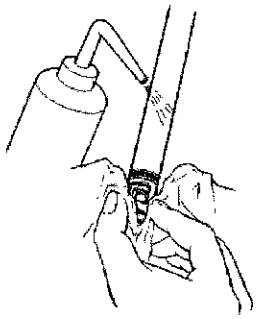
● Clearing calibrated values

To clear all the calibrated values:

1. Set the pH meter to the Calibration mode.
2. Press the CAL key while holding the SET key down.

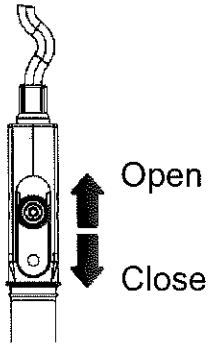
● Measuring pH

1. Wash the tip of electrode well with pure (de-ionized) water, and then wipe with filter paper or tissue paper.



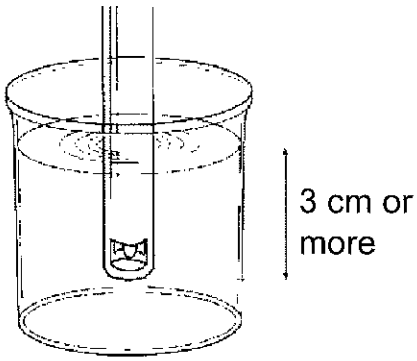
2. Open the internal solution filler port.

Leave the port open while measurement is taking place.



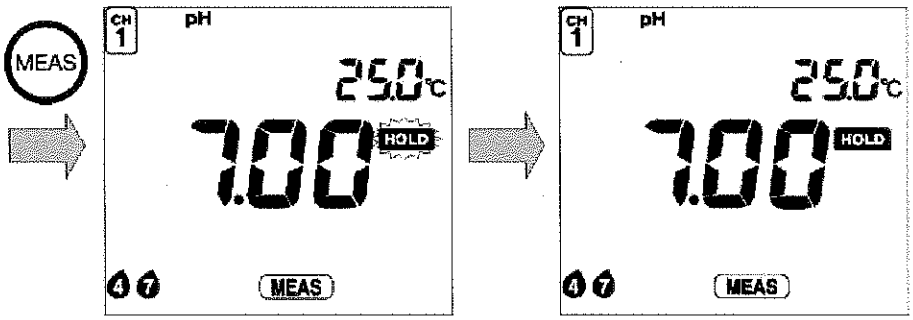
3. Immerse the electrode all the way in the sample.

Immerse the pH electrode in the sample at least three centimeters.



4. Press the MEAS key with the initial screeen displayed.

"HOLD" will blink until the reading stabilizes.
When the indicated value stabilizes, "HOLD" will stop blinking and will be displayed. The indicated value will remain displayed continually.



Ref.

Refer to the " Criteria for judging stability" page 17 for the criteria for judging the stability of the readout.

Note

When measurement data is held using Instantaneous Value Measurement or Auto Hold Measurement, you can store that data in the memory by pressing the ▲ key. See "3.1 Data memory function" page 29.

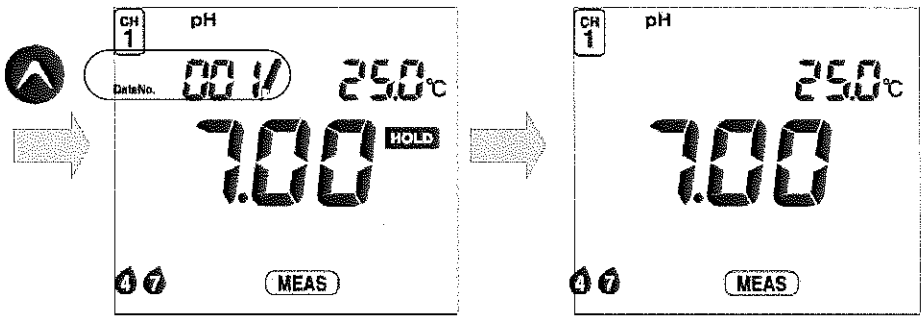
3 Functions

This chapter describes the various functions of the pH meter.

3.1 Data memory function

● Data memory

In all measurement modes, you can store data when the instantaneous value is measured or the measured value is held (HOLD status) during the Auto HOLD measurement by pressing the ▲ key.
The measurement reading is stored along with the temperature, data, HOLD value/instantaneous value, ATC/MTC and calibration point , at the time the measurement was taken.
After the data number is displayed, the screen returns to the initial screeen. Up to 300 items of data can be stored in the memory. If the number of data items exceeds the maximum limit, ERR 10 is displayed and no more data can be stored.

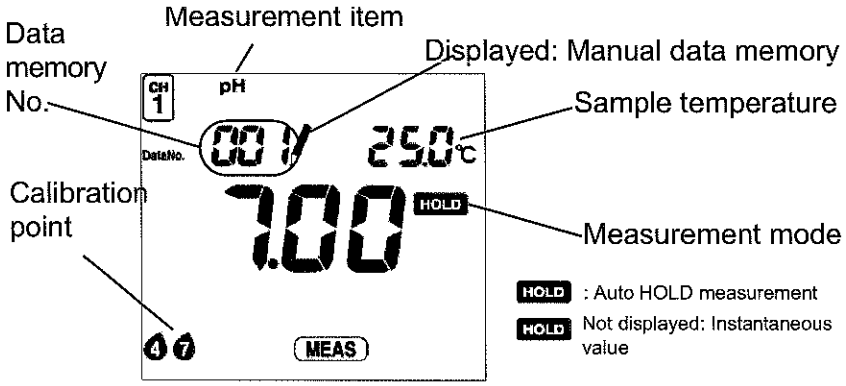


Note

Data cannot be stored unless the value has stabilized or in the CAL mode.

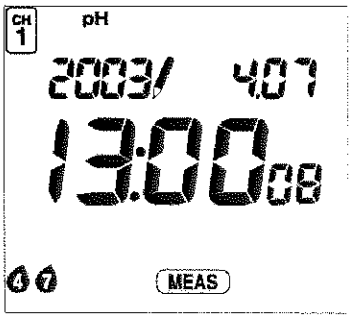
● Calling up memory data

1. Press the ▼ key in the Measurement mode to load measurement data.



Select and load the desired memory data item using the ▲ and ▼ keys. The displayed number returns to 0 after 300, the maximum number.

2. Press the MODE key to display the data and time.



Select the desired data item using the ▲ and ▼ keys.

3. Press the MODE key to display the ID.

Select the desired data item using the ▲ and ▼ keys.

Note

If an error occurs while a data number is being displayed, the error number will NOT be displayed. When using a printer (sold separately), press the ENTER key while in the DATA OUT mode to print the data.

3.2 pH calibration history display

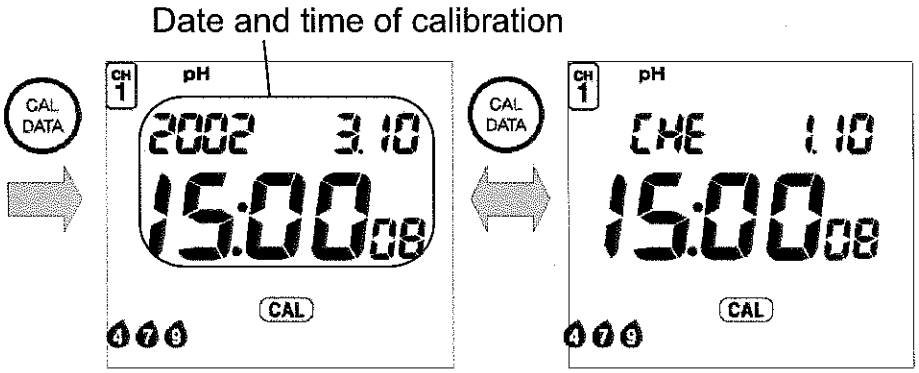
The latest calibration and repeatability check information can be checked.

Ref.

Refer to "pH repeatability check" on page 26.

● pH calibration history

1. Press the CAL DATA key in the pH Measurement mode.



Latest calibration date

Latest check date

2. Pressing the CAL DATA key toggles between the latest calibration date and the latest check date.

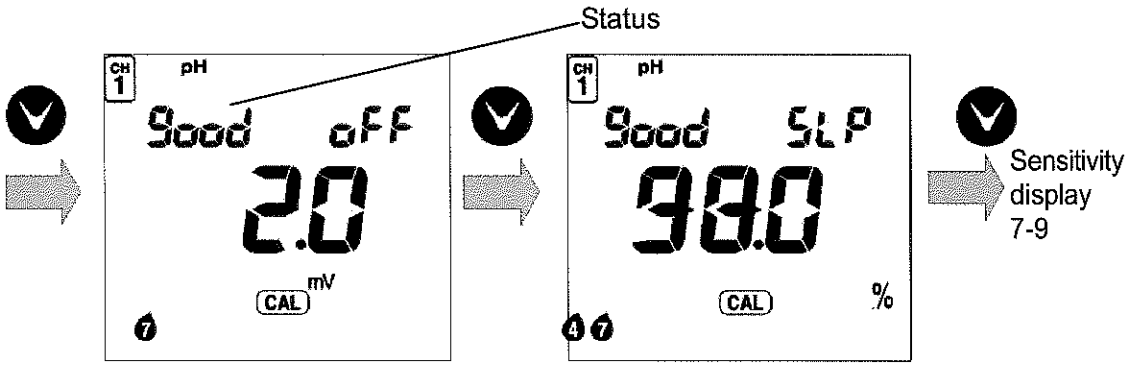
Note

The latest check date is not displayed if no repeatability check has been performed.

Latest calibration data

1. Press the ▼ key with latest calibration date displayed.

The asymmetrical potential will be displayed.



Asymmetrical potential display Sensitivity display 4-7

Example of 3-point calibration

2. Press the ▼ key to show sensitivity display.

Status display

Good	When the meter is in a good condition
CHE	When the electrode needs washing
bAd	When the electrode is old and is going bad

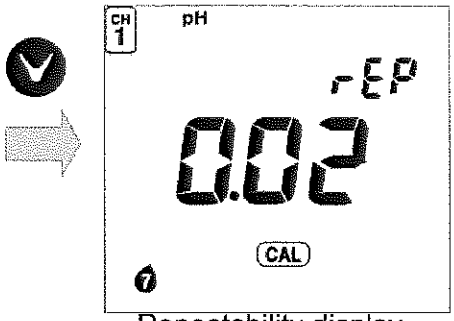
Ref.

Refer to "Asymmetrical potential display" on page 71.

pH latest check data

1. Press the ▼ key with latest check date displayed.

The repeatability display will appear.



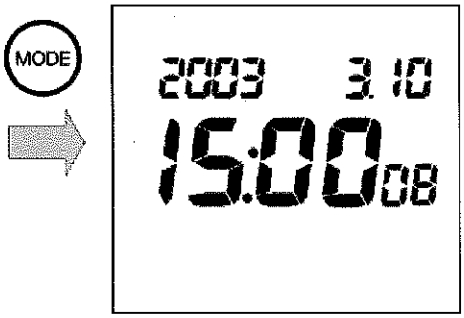
Repeatability display

3.3 Displaying and setting the clock

The clock needs to be when the meter is used for the first time or after replacing the batteries.

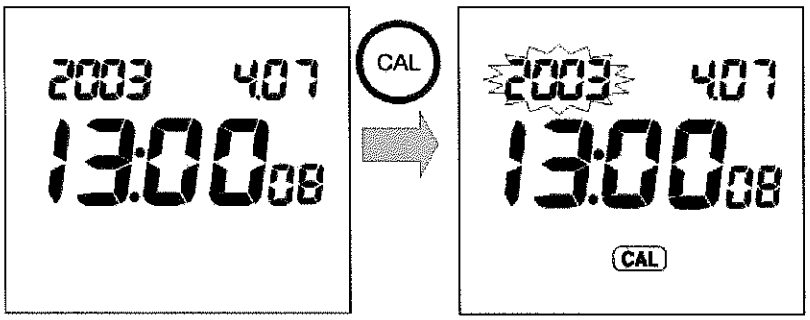
● Displaying the clock

Press the MODE key in the Measurement mode to display the clock.



● Setting the clock

1. Press the CAL key when the Clock Display screen is displayed to show the Setting screen for the clock.



2. Switch the display to year, month, day, hour, minute, and second using the ENTER key. You can set a numerical value using the ▲ and ▼ keys.

Note

Set the seconds to "00" sec. Pressing the ENTER key sets it to "00".

3. After setting the clock, press the ENTER key to update the setting.

   Pressing the CAL key at this time returns you to the Clock Display screen without changing the current setting.

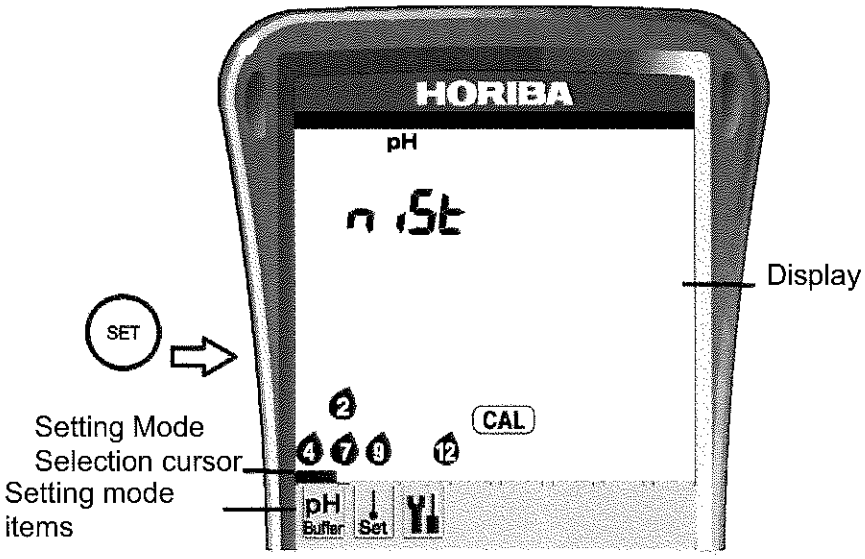
4. Press the MODE key to return to the Measurement mode.

3.4 Setting modes

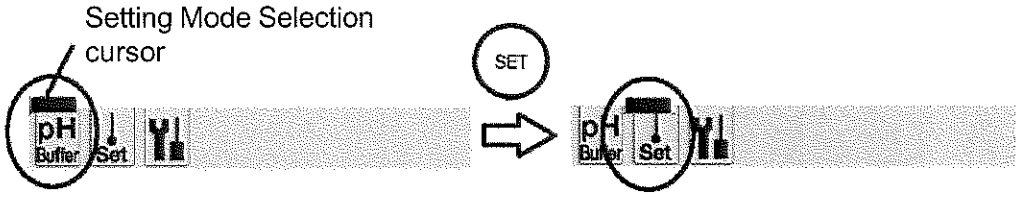
Selecting the Setting mode expands the uses of the meter.

3.4.1 Entering the Setting mode

1. Press the SET key in the Measurement mode. The Setting Mode Selection cursor appears at the left-bottom of the screen to indicate that the Setting mode is active.



2. Pressing the SET key moves the Setting Mode Selection cursor one by one to allow you to select the Setting mode of your choice.



3. Press the MEAS key to return to the Measurement mode from the Setting mode.

3.4.2 Display and description

Display	Name	Description	Page No.
pH Buffer	pH Buffer Setting	Sets the standard solution for pH calibration.	page 37
Set	Temperature Compensation Setting	Selects the Auto/Manual mode for temperature compensation.	page 41
	Maintenance	Sets various maintenance-related settings.	page 42

3.4.3 pH standard solution setting

The meter allows you to select the standard solution specifications used for calibration from among the NIST standard, US standard (USA), and use-defined standard (CUST).

NIST standard	When using the standard solution required by NIST standards
	Bottle mark ❷ ❹ ❼ ❾ ⓬
US standard (USA)	When using the standard solution required by US standards
	Bottle mark ❷ ❹ ❼ ❿ ⓬
Custom (CUST)	When using a user-defined standard solution
	Bottle mark ❶ ❷ ❸

Ref.

Refer to "Types of pH standard solution" on "● Types of pH standard solutions" page 72.

Note

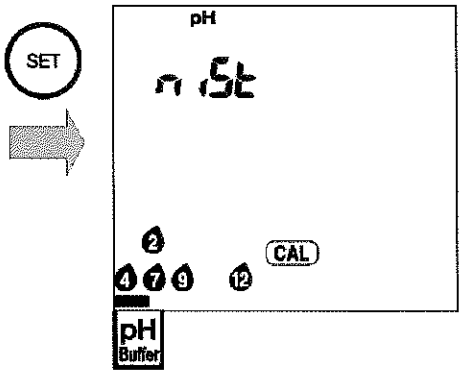
The calibrated value for pH 7 standard solution is different between the NIST standards and US standards.

NIST standard: pH 6.86 (at 25°C)

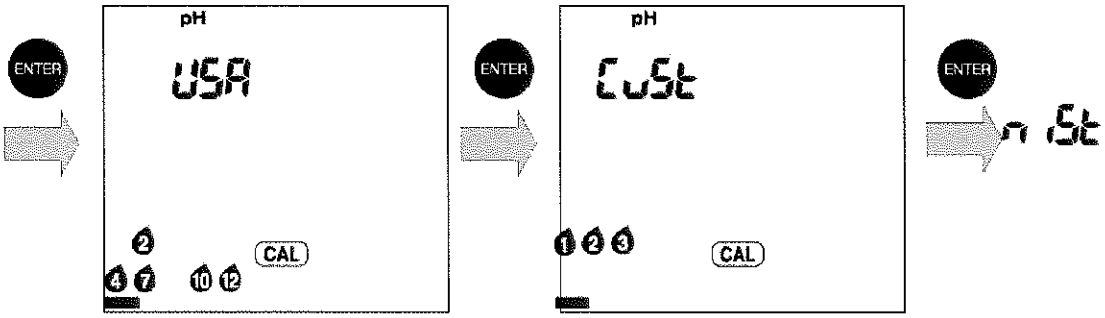
US standard: pH 7.00 (at 25°C)

Changing the standard solution setting

1. Press the SET key in the Measurement mode and select the pH Buffer Setting mode.



2. Press the ENTER key to toggle between NIST standard, US standard (USA) and a user-defined standard (CUST).



3. Press the MEAS key to return to the Measurement mode.

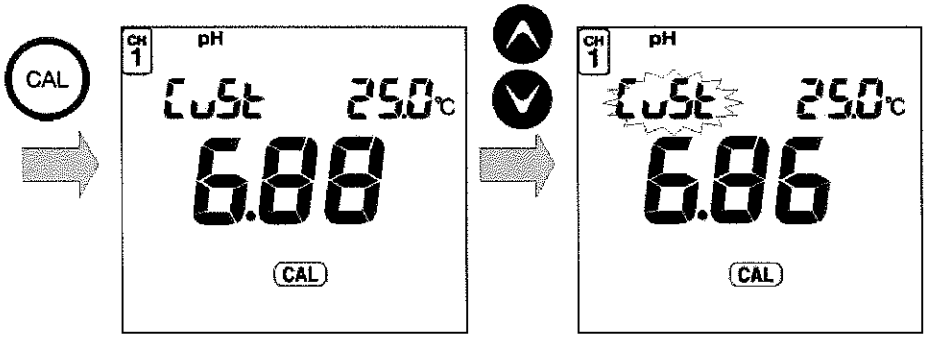
Calibration using a user-defined standard (CUST)

1. Press the CAL key in the pH Measurement mode to select the Calibration mode.

“>CAL<“ will be displayed.

2. Set the pH value of the standard solution used for calibration using the ▲ and ▼ keys.

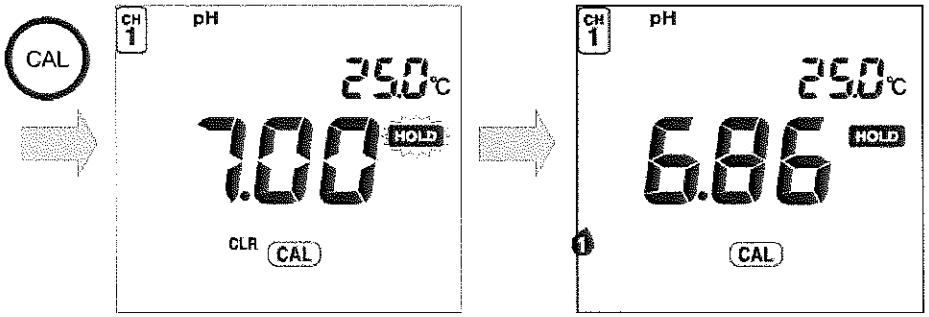
While the setting is being made, “CuSt” will blink.



3. Press the CAL key to start the calibration.

The measured value will be displayed, and “HOLD” will blink until the reading stabilizes.

When the measured value stabilizes, “HOLD” will stop blinking and the calibrated value will be displayed.

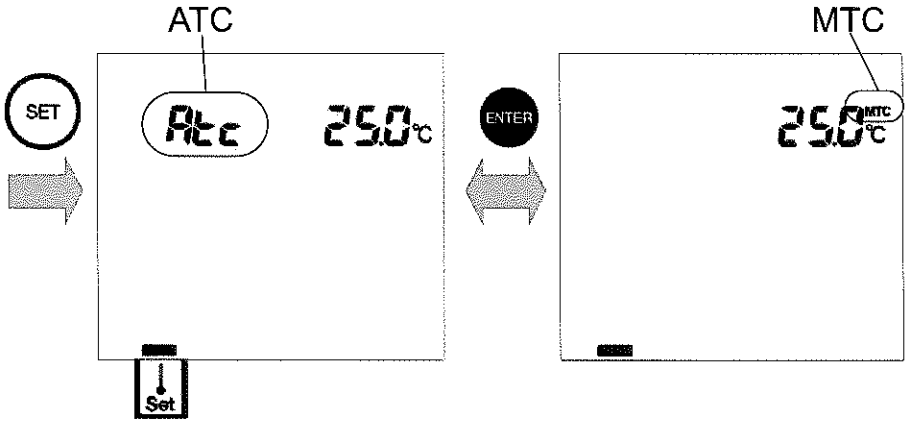


Note

Perform the calibration for the second and third point following the same procedure.

3.4.4 Temperature compensation setting

1. Press the SET key in the Measurement mode to enter the Temperature Compensation Setting mode.
2. Pressing the ENTER key toggles between MTC and ATC settings.

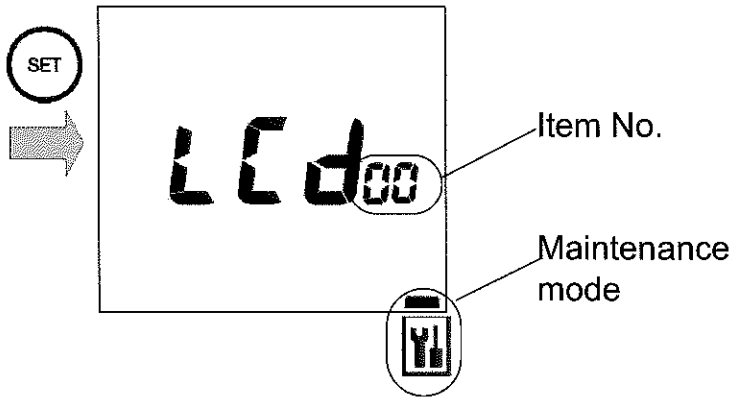


ATC
Automatic temperature compensation (when using a temperature sensor of the electrode)
ATC is displayed.
When a temperature sensor is connected, the current temperature is automatically displayed.
(When no temperature sensor is connected, the display shows 25°C.)

MTC
Manual temperature compensation (when an electrode temperature sensor is not being used and the temperature of the solution is known before hand)
MTC is displayed.
Set the temperature using the ▲ and ▼ keys.
Setting range: 0.0 to 100.0°C

3.4.5 Maintenance mode

Press the SET key in the Measurement mode and select the Maintenance mode. The LCD CHECK screen (Item No. 00) will appear.



● Maintenance setting items

Use the MODE key to toggle between Maintenance mode items.

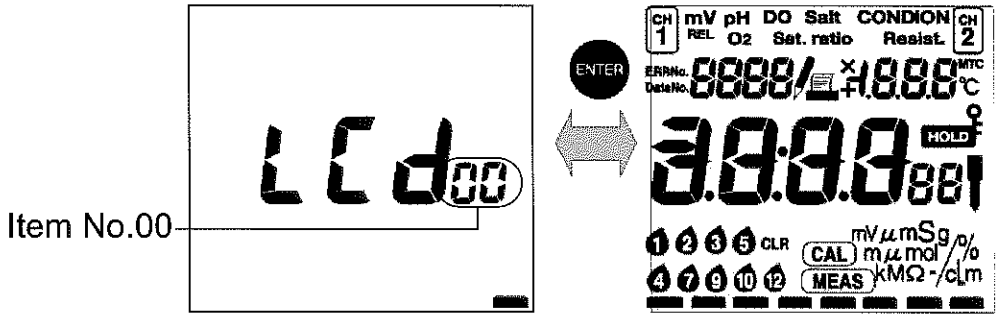
Item No.	Item	Description	Page No.
00	LCD check	Enables check to see if all LCD segments are displayed.	page 44
01	Battery voltage check	Enables simple battery voltage check.	page 45
02	Temperature zero adjustment	Carries out temperature calibration when the temperature sensor is immersed in a liquid of known temperature.	page 46
03	Automatic power-off setting	Turns Automatic Power-off function ON/OFF and sets time period after which the power will be turned off when no keys are touched.	page 47
05	Remaining data memoriy	Displays number of data items that can still be stored.	page 48
06	Data memory clear	Clears all data in the data memory.	page 48
07	Initialization of setting	Initializes all settings to default values.	page 49

● LCD check [item No. 00]

Displays all segments of the LCD.

1. Press the MODE key in the Maintenance mode to show item No. 00.
2. Press the ENTER key.

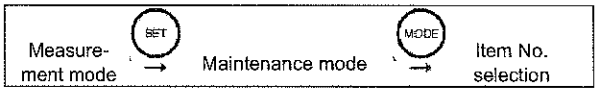
   Compare the LCD screen with this diagram to confirm that all segments of the LCD are displayed.



3. Use the MODE key to proceed to the Battery voltage check (item No. 01).

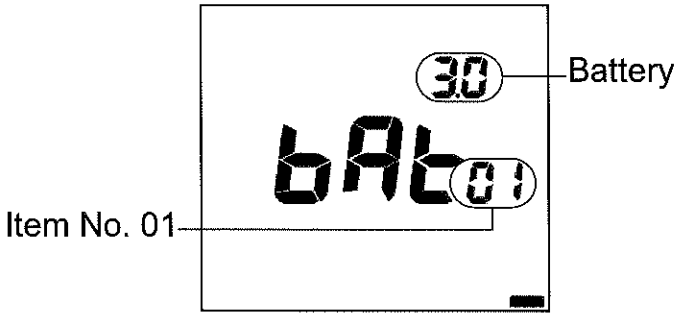
— HINT! —

Entering the Maintenance mode



● Battery voltage check [item No. 01]

The battery voltage (V) is displayed.



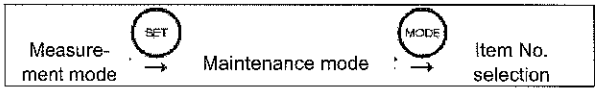
Note

The battery voltage alarm is set at approximately 2.2 V. The measured voltage for batteries depends on the current. The voltage shown in this mode will be a little lower than the actual voltage.

1. Use the MODE key to proceed to temperature zero adjustment (item No. 02).

HINT!

Entering the Maintenance mode



● Temperature zero adjustment [item No. 02]

This mode uses a known temperature to calibrate the temperature compensation value. This mode is used when calibrating the temperature of the thermometer.

1. Immerse the electrode in a liquid with a known temperature, and set the temperature using the ▲ and ▼ keys.

Setting range: 0.0 to 100.0 °C

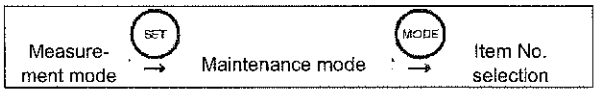
Note

The temperature sensor attached to the electrode maintains an accuracy of ±1°C, even without calibration. The above mode should be used when a greater precision than ±1°C is required.

2. Use the MODE key to proceed to Automatic power-off setting (item No. 03).

HINT!

Entering the Maintenance mode



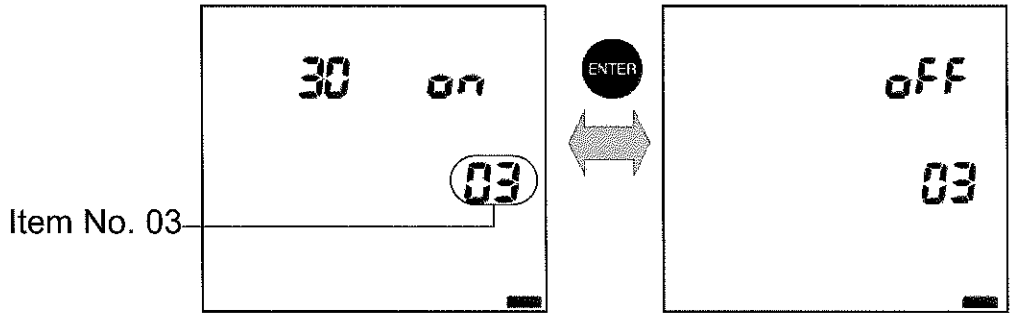
● Automatic power-off setting [item No. 03]

This turns the Automatic Power-off function ON/OFF and sets the time until the power is turned off.
When the Automatic Power-off function is set to ON, the power to the meter automatically turns off if the keys are not operated for the set amount of time.

1. Press the ENTER key to toggle between ON and OFF.

When set to ON, set the time for the power to be turned OFF using the ▲ and ▼ keys.

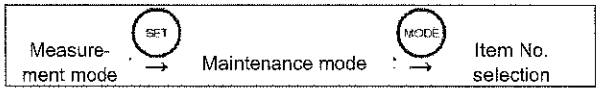
Setting range: 1 to 30 minutes



2. Press the MODE key to proceed to [Item No. 05] Remaining data memory.

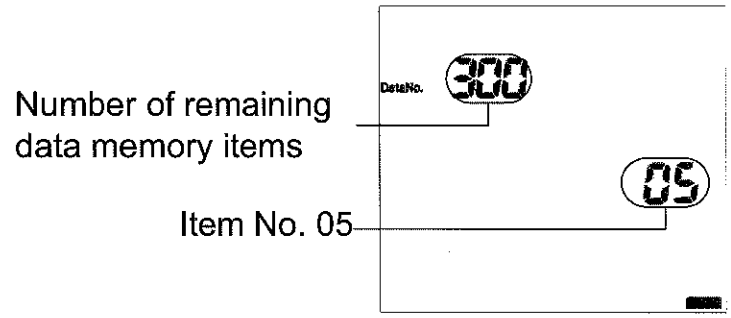
— HINT! —

Entering the Maintenance mode



● Remaining data memory [Item No. 05]

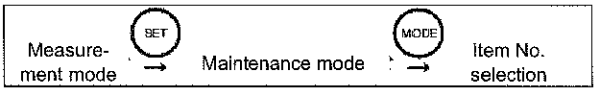
Displays the number of data items that can still be stored.



1. Press the MODE key to proceed to Data memory clear (item No. 06).

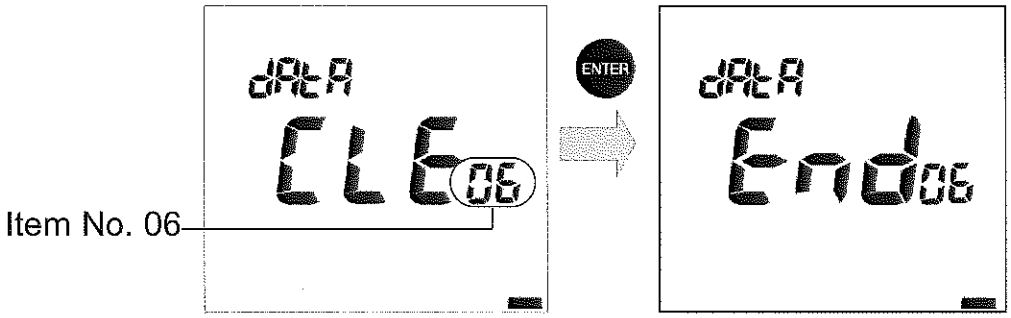
— HINT! —

Entering the Maintenance mode



● Data memory clear [Item No. 06]

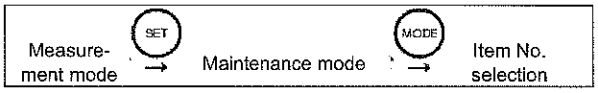
1. Pressing the ENTER key clears all the data stored in the memory.



2. Press the MODE key to proceed to Initialization of setting (item No. 07).

— HINT! —

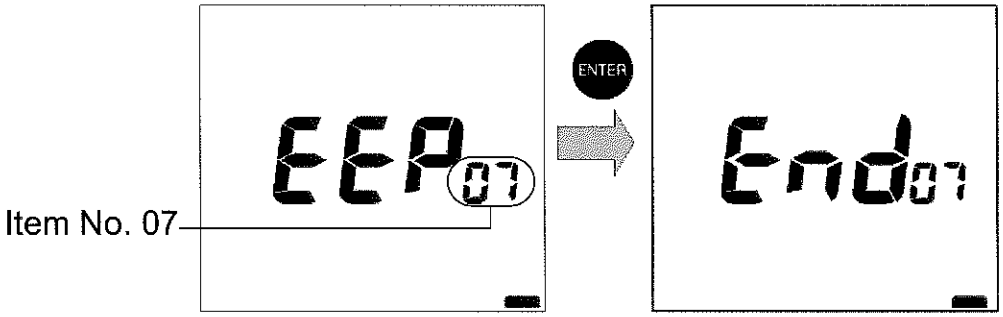
Entering the Maintenance mode



● Initialization of setting [item No. 07]

This mode returns all settings to the default settings. Use this mode to return the pH meter to the original settings when the meter was purchased.

1. Press the ENTER key to initialized the settings.

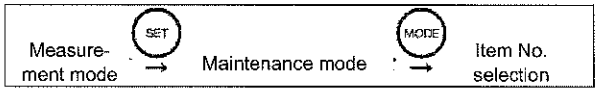


— Ref. —

The setting values to be initialized are shown on page 77.

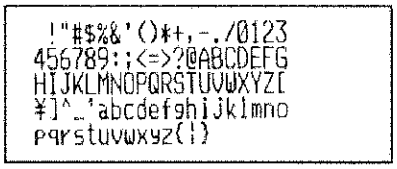
— HINT! —

Entering the Maintenance mode



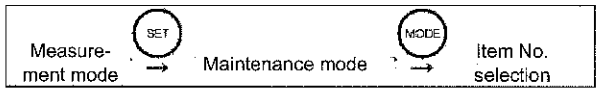
2. Press the MODE key to return to the first item in the maintenance modes, LCD check.

Test print format



— HINT! —

Entering the Maintenance mode



4 Maintenance and Troubleshooting

This chapter explains how to perform daily meter maintenance and how to deal with error messages.
Daily maintenance is vital in assuring accurate measurement and preventing breakdowns before they occur. Maintenance of the electrodes is especially important; if ignored, various problems and erroneous measurements may result. This meter is equipped with a convenient error message function. If an error message is displayed, be sure to take appropriate action.

4.1 pH electrode maintenance

Maintain your electrodes by referring to the following information or to the operation manuals for the electrodes.

Injury warning

Caution

Glass fragments can cause injury.
The outer tube of the electrode and the tip of the electrode are made of glass.
Use care not to break them.

Maintenance after daily use

After taking measurements, wash the electrode using pure water (de-ionized water), wipe off the water from the electrode with filter paper or tissue paper, and store it with its cap on.

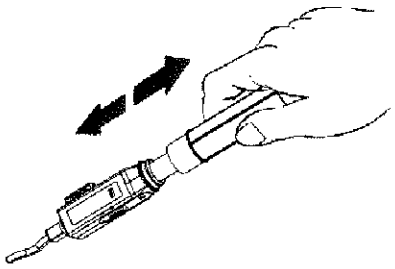
Note

The liquid junction may become clogged if the electrode is left in distilled water.

● Extended storage

When an electrode is not to be used for a long period of time, store the electrode after performing the following steps. Also, replace the reference solution every three to six months, using the method explained below.

1. Remove the electrode from the pH meter.
2. Remove the protective cap from the electrode.

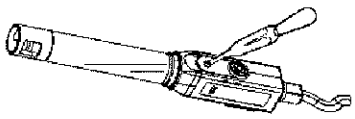


Chemical warning

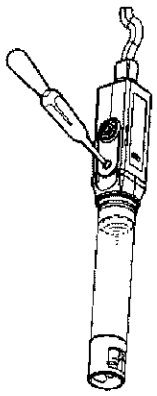
Caution

The liquid inside the electrode is highly concentrated potassium chloride (3.33 mol/L KCl).
If the internal solution in the electrode comes in contact with your hands or skin, wash immediately with water. If the internal solution comes in contact with your eyes, flush immediately with large amounts of water and seek treatment by a physician.

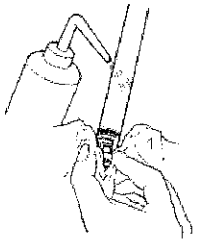
3. Open the internal solution filler port and use a syringe to remove the internal solution.



4. Fill the electrode with new internal solution (#300), until it nears the opening.



5. Wash the tip of the electrode well with pure (de-ionized) water and wipe it with filter paper or tissue paper.



6. If the liquid on the inside of the electrode cap has dried, wash the inside of the electrode cap with pure (de-ionized) water, and then, after shaking out the water, fill the cap with enough pure water to soak the sponge.

Note

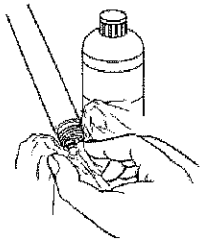
If the solution inside the protective cap for the electrode has dried up and the electrode has not been used for an extended period of time, the response speed of the electrode may be slower (and its sensitivity lower) than before.

● Washing the electrodes

If the tip of the pH electrode is extremely dirty, the speed of its response may slow and it may cause errors in measurement. If the electrode is so dirty that it cannot be cleaned by rinsing with pure (de-ionized) water, wash the electrode using the most appropriate method below.

General dirt & oily grime

Wipe the dirt/grime off using cotton gauze that contains a neutral detergent.



Inorganic grime

Rinse using a hydrochloric acid solution or cleaning liquid (#220) of approximately 1 mol/L. Be sure not to soak the electrode in strong acid for a long period of time.

4.2 Troubleshooting

The meter is equipped with a simply error-message function to notify the operator that an operation error or problem with the equipment has occurred. Errors or other problems that occur while in the Measurement mode are announced by an error No. appearing in the lower left-hand corner of the display.

4.2.1 Error message chart

ERR No.	Message	Explanation
01	Memory error	Data cannot be read from or written to the internal memory.
02	Battery voltage low	The battery voltage is low.
03	Electrode stability error	The electric potential did not stabilize within three minutes.
04	Asymmetric potential error	pH: The asymmetric potential of the electrode is 45 mV or more.
05	Electrode sensitivity error	pH: The electrode sensitivity is either 105% or more or 85% or less than the theoretical sensitivity. DO: The electrode sensitivity is out of the standard.
06	Max. calibration points exceeded	pH: No more than three points can be calibrated.
07	Cannot identify standard solution	pH: The pH meter cannot identify the standard buffer.
10	Data memory over	The number of data items has exceeded the limit of the memory.

● ERR No. 01 Memory error

Explanation

Data cannot be read from or written to the internal memory.

Cause	How to solve problem
The pH meter does not start operating correctly even after the power is turned ON.	Take the battery from the pH meter, and disconnect the AC adapter. Then press the ON/OFF key for about 10 seconds.
The internal IC is defective.	Seek repairs at your nearest retail outlet or HORIBA service station.

● ERR No. 02 Battery voltage low

Explanation

The battery has insufficient voltage.

Cause	How to solve problem
The battery voltage is low. (Battery voltage: 2.2V or less)	Replace the dry-cell battery.

Note

The measured value cannot be guaranteed when ERR No. 02 is displayed.

● ERR No.03 Electrode stability error

Explanation

The electric potential did not stabilize within three minutes.

Cause	How to solve problem
This is caused by the sample solution (when the sample solution is pure water or another solution with low conductivity or the pH concentration or temperature change).	Press the MEAS key again while "HOLD" is either brinking or steadily lit in the display, to measure the sample using instantaneous value measurement.
The electrode is dirty.	Wash the electrode.
The electrode is cracked.	Replace the electrode.
The responsive glass membrane of the electrode has been dry for a long time.	Soak the membrane (on the electrode) in pure (de-ionized) water for 24 hours.
The temperature of the sample solution is fluctuating.	Measure after the sample solution temperature stabilizes.

● ERR No.04 Asymmetric potential error

Explanation

The asymmetric potential of the electrode is 45 mV or more.

Cause	How to solve problem
The electrode is dirty.	Wash the electrode.
The electrode is cracked.	Replace the electrode.
The reference solution concentration is fluctuating.	Replace the internal solution in the reference electrode.
The electrode is not connected correctly.	Connect the electrode correctly.
The electrode is not submerged deeply enough to cover the liquid junction (tip).	Immerse the electrode in the sample at least three centimeters deep.
There is problem with the standard solution.	Prepare new standard solution.

● ERR No.05 Electrode sensitivity error (pH)

Explanation

The electrode sensitivity is either 105% or more or 85% or less than the theoretical sensitivity.

Cause	How to solve problem
The electrode is dirty.	Wash the electrode.
The electrode is cracked.	Replace the electrode.
Calibration was not performed correctly.	Redo the calibration correctly.
There is a problem with the standard solution.	Use fresh standard solution.
The electrode is not connected correctly.	Connect the electrode correctly.
Electrode is not submerged deeply enough to cover reference junction.	Immerse the electrode in the sample at least three centimeters deep.

● ERR No.06 Max. calibration points exceeded

Explanation

Calibration was performed on a fourth calibration point.

Cause	How to solve problem
Calibration was performed on a fourth calibration point.	Limit the number of calibration points to three. This error message is cleared by setting the pH meter to Measurement mode.

Note

This error does not affect calibration data obtained from previous three calibration points.

Ref.

Refer to " ● Standard solution calibration" page 21.

● ERR No.07 Cannot identify standard solution

Explanation

If the automatic standard-solution identification function of the meter does not work, recalibrate the meter after performing the appropriate measures below.

Cause	How to solve problem
There is a problem with the standard solution.	Prepare new standard solution.
There is a problem with the standard solution setting.	Check the NIST or US standards settings and the kind of standard solution used for calibration, and make sure they match.
The responsive membrane is dry or dirty.	Measure after washing the responsive membrane and soaking it in pure (de-ionized) water for 24 hours.
The reference solution is contaminated.	Replace the reference solution with new solution.
The responsive membrane is damaged or worn out.	Replace the electrode.

● ERR No.10 Data memory over

Explanation

The number of data items has exceeded the limit of the memory.

Cause	How to solve problem
Memory over	Delete data stored in the memory after confirming their contents.

4.2.2 More troubleshooting

This section explains how to respond to various symptoms of trouble that are not indicated by an error number.

● Nothing shows up on the display when the power is turned ON

Cause	How to solve problem
No batteries	Place batteries in the meter.
The batteries are loaded with the poles reversed.	Re-insert the batteries with the poles correctly oriented.
The battery voltage is low.	Remove the old batteries and correctly insert new dry-cell batteries. Or connect the unit to the optional AC adapter.

● The indicated value fluctuates

When there is a problem with the electrode...

Cause	How to solve problem
The responsive membrane is dry or dirty.	Wash the responsive membrane.
The responsive membrane is damaged or worn out.	Replace the electrode.
There are air bubbles on the electrode.	Shake the electrode to remove the air bubbles.
There is no reference solution remaining.	Fill the electrode with new reference solution, as noted in the electrode operation manual.
The wrong reference solution is being used.	Use the correct reference solution.

When there is a problem with the main unit of the pH meter...

Cause	How to solve problem
There is a motor or other device causing electrical interference.	Move the meter to a place where it is not subject to dielectric effects. Be sure to ground devices that are using commercial electricity.
The electrode is not connected correctly.	Connect the electrode correctly.

When there is a problem with the sample solution...

Cause	How to solve problem
The liquid junction is not immersed in the sample solution.	Immerse the electrode in the sample solution up until the liquid junction or deeper.
Some effects of the sample	Determine if this is the cause by measuring with a stable standard solution.

● The response is slow

Cause	How to solve problem
Some effects of the sample	Response time may slow down, depending on the properties of the sample solution.
The electrode is dry or dirty.	Wash the responsive membrane.
The electrode is cracked or worn out.	Replace the electrode.
There is a problem with the reference solution.	Fill the electrode with new reference solution, as noted in the electrode instruction manual.

● The indicated value does not change, or there is absolutely no response

Cause	How to solve problem
The system is locked.	Turn the power OFF, and then turn it back ON again.
The electrode connector is not attached correctly.	Attach the electrode connector correctly.
pH meter is defective.	Contact your local HORIBA distributor.

● The measured value is blinking

The pH value exceeds the measurement range (when pH value is displayed).

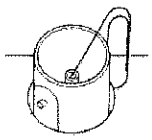
Measurement range: pH 0.00 – pH 14.00

Cause	How to solve problem
The sample solution is inappropriate.	Change to a sample solution with properties within the measurement range.
The liquid junction is not immersed in the sample solution.	Immerse the electrode in the sample solution all the way until the liquid junction or deeper.
The electrode cable has been severed.	Replace the electrode.
The main body of the pH meter is defective.	Check the point described below.
The meter has not been calibrated or it has been calibrated incorrectly.	Calibrate the meter correctly.

Check this point

As shown in the diagram, use a jumper wire or bent paper clip to short the meter by touching both the center pin and some metal part in the electrode connector.

If the flaching measured value disappears when this done, the meter is nomal.



● The temperature display is blinking. The temperature display does not change from 25°C.

The temperature measurement exceeds the measurement range.

Measurement range: -10 – 100.0°C

Cause	How to solve problem
The temperature of the sample solution exceeds the measurement range.	Check the temperature of the sample solution and change to a sample solution that has a temperature within the measurement range.
The thermistor connection within the electrode is severed or shorted.	Measure the resistance of the temperature sensor connector. If it is 50 kΩ or more at room temperature, replace the electrode.
The electrode connector is not attached properly.	Attach the electrode connector properly, so that the O-ring on the temperature connector disappears from sight.
The main unit of the meter may be defective.	In Temperature Display Calibration mode (See " ● Temperature zero adjustment [item No. 02]" page 46), check whether or not the "Minus" display appears, regardless of whether or not there is a temperature connector.
There is a problem with the setting for the temperature display calibration mode (see page 46).	Initialize the settings (see page 49).

● Measurements are not repeatable

Cause	How to solve problem
Some effects of the sample solution	The pH or other properties of the sample solution may have changed over time, making repeatability poor.
The responsive membrane is dry or dirty.	Wash the responsive membrane.
There is not enough reference solution or it is dirty.	Replace the reference solution with new solution.
The responsive membrane is cracked or worn out.	Replace the electrode.

5 Reference

This chapter provides a simple compilation of information for those who would like to know about the functions of the main unit of the meter and other measurement principles in greater detail.
It also serves as a reference for spare and optional parts.

5.1 pH measurement

● pH measurement and temperature

The temperature of the solution being inspected is an important parameter in the accurate measurement of pH. There are many possible sources of errors during measurement, such as the state of the solution junction potential, asymmetric potential, and reference solution pH concentration, but all of these items contain factors that change with temperature. The best way to minimize these potential causes of errors is to keep the temperature of the pH standard solution uniform at the time of calibration.

● Liquid junction potential

"Liquid junction potential" is the electric potential that occurs to a greater or lesser degree at the liquid junction. The size of the electric potential differs depending on the type of solution, the temperature of the solution, and the structure of the liquid junction.

When solutions of different compositions come in contact, ION diffusion occurs on the contact surface between the two solutions. The ions are of various sizes, so a difference occurs in the diffusion transfer speed.

As diffusion proceeds, a difference in charges occurs on the contact surface of the two solutions, giving rise to a difference in potential. This potential works to reduce the transfer speed of fast ions and increase the speed of slow ions, ultimately achieving a state of equilibrium when the transfer speed of the positive and negative ions on the contact surface of the two solutions is equal. In this state of equilibrium, the potential at the contact surface between the two solutions is called the "liquid junction potential." A large liquid junction potential means measurements will be very inaccurate.

● Asymmetric potential

The glass electrode is immersed in a pH 7 reference solution. When the electrode is immersed in the pH 7 solution, both the internal and external sides of the electrode membrane are supposed to take on a pH of 7, making the potential 0. In actuality, however, a potential does occur. This potential is called "asymmetric potential." The size of the asymmetric potential differs depending on any stress that may have occurred during the processing of the glass and the shape and compositions of the glass. Asymmetric potential also changes depending on the degree of contamination of the reference solution and the state of the glass membrane. Also, if the electrode membrane dries out, a large asymmetric potential will occur, giving rise to measurement errors.

● Temperature compensation

The electromotive force generated by the glass electrode changes depending on the temperature of the solution. "Temperature compensation" is used to compensate for the change in electromotive forces caused by temperature. There is absolutely no relation between the change in pH caused by the temperature of the solution and temperature compensation. This is often misunderstood. When pH is to be measured, the temperature of the solution when the pH is measured must be recorded along with that pH value, even if a meter that has automatic temperature compensation is used. If the solution temperature is not recorded, the results of the pH measurement are relatively meaningless.

● Types of pH standard solutions

When measuring pH, the pH meter must be calibrated using a standard solution. There are several kinds of standard solutions. For normal measurement, three standard solutions—with a pH of 4, 7, and 9—are sufficient to accurately calibrate the meter.

- pH 1.68 standard solution: Oxalate
  0.05 mol/L tetra-potassium oxalate aqueous solution
- pH 4.00 standard solution: Phthalate
  0.05 mol/L potassium hydrogen phthalate aqueous solution
- pH 6.86 standard solution: Neutral phosphate
  0.025 mol/L potassium dihydrogen phosphate, 0.025 mol/L sodium dihydrogenphosphate aqueous solution
- pH 9.18 standard solution: Borate
  0.01 mol/L tetra-sodium boric acid (boric sand) aqueous solution
- pH 12.45 standard solution: Saturated calcium hydroxide solution

pH values of pH standard solutions at various temperatures (NIST (former NBS) settings)

Temp. (°C)	pH 1.68 standard solution Oxalate	pH 4.00 standard solution Phthalate	pH 6.86 standard solution Neutral phosphate	pH 9.18 standard solution Borate	pH 12.45 standard solution Saturated calcium hydroxide solution
0	1.666	4.003	6.984	9.464	13.423
5	1.668	3.999	6.951	9.395	13.207
10	1.670	3.998	6.923	9.332	13.003
15	1.672	3.999	6.900	9.276	12.810
20	1.675	4.002	6.881	9.225	12.627
25	1.679	4.008	6.865	9.180	12.454
30	1.683	4.015	6.853	9.139	12.289
35	1.688	4.024	6.844	9.102	12.133
38	1.691	4.030	6.840	9.081	12.043
40	1.694	4.035	6.838	9.068	11.984
45	1.700	4.047	6.834	9.038	11.841

Note

When the standard solutions use US settings, the pH 7 values shown in the following table are different and pH 9 becomes pH 10 (see next page).

pH values of pH 7 and pH 10 standard solutions at various temperatures (US-standard settings)

Temp. (°C)	pH 7 standard solution Neutral phosphate	pH 10 standard solution Carbonate
0	7.119	10.318
5	7.086	10.245
10	7.058	10.178
15	7.035	10.117
20	7.015	10.061
25	7.000	10.011
30	6.988	9.965
35	6.979	9.925
40	6.973	9.888
45	6.969	9.856

Note

Calibration is performed using Nernst's equation with the above values.

● Using standard solutions

Standard solutions are used to calibrate the scale of the pH meter employed to measure the unknown pH of a solution. Standard solutions of pH 4, 7, and 9 are used in combination according to the particular conditions of the solution that is to be inspected.

When the approximate pH value is desired (1-point calibration)

Use the pH 7 standard solution or a standard solution that approximates the pH value of the solution that is to be inspected.

When it is known beforehand whether the test solution is acidic or alkaline (2-point calibration)

Acidic: Use the pH 4 and 7 standard solutions.
Alkaline: Use the pH 7 and 9 standard solutions.

When an unknown solution is to be inspected (3-point calibration)

Use the pH 4, 7, and 9 standard solutions.

Other

When finding the pH of other solutions, perform 2-point or 3-point calibration using pH 2, 4, 7, 9, or 12 standard solutions randomly, then measure the test solution.

5.2 Specifications

● Measurement target

Target	Item	Description
pH	Measurement principle	Glass electrode
	Display range	pH -2.00 – 16.00
	Measurement range	pH 0.00 – 14.00
	Resolution	0.01 pH
	Repeatability	±0.01 pH ±1digit
Temp.	Measurement principle	Thermistor
	Measurement range	0.0 – 100.0 °C
	Resolution	0.1 °C
	Repeatability	±0.1 °C ±1digit

● Items in common among meter models

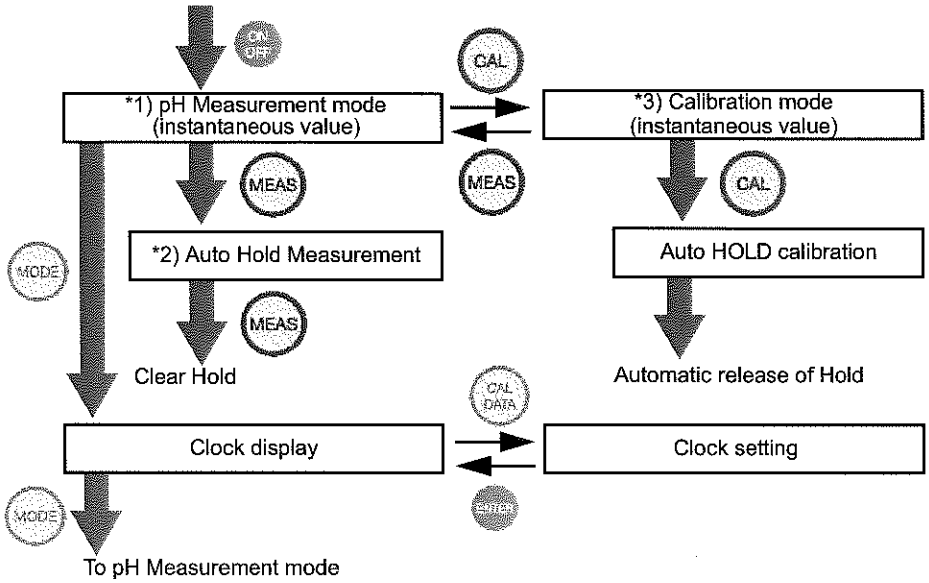
Data memory capacity	Max. 300 pieces of data
Power	Dry cell batteries type:AA alkaline with automatic power OFF function
Ambient temperature	0 – 45 °C
Dimensions	170(H) × 80(W) × 40(D) mm
Mass of main unit (including batteries)	300 g

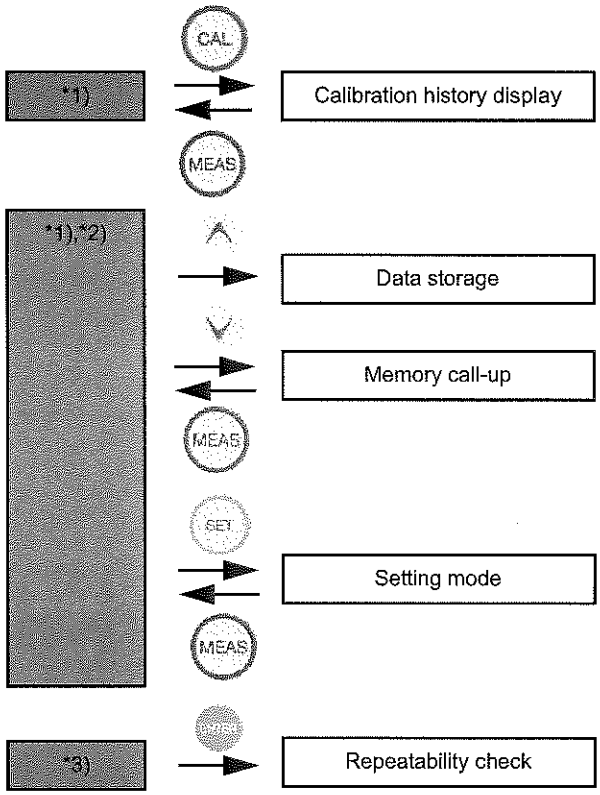
5.3 Default settings

Category	Item	Default values
Common setting	Temperature compensation	Automatic temperature compensation
	Manual temperature compensation	25 °C
	Automatic power OFF	Approx. 30 min (ON)
pH	Standard calibration solution	NIST
	Calibration setting	Asymmetric potential: 0mV Sensitivity: 100%

5.4 Operation flowcharts

The following flowcharts summarize pH meter operations





5.5 Spare and optional parts

This section lists spare and optional parts for the pH meter.
These parts are available through HORIBA distributors. Place an order specifying their name, model, and part number.

5.5.1 Spare parts list

● pH electrode (with built-in temperature sensor)

Part name	Model	Part number	Remarks
D-50 series standard electrode	9621-10D	9096001700	Plastic-body electrode (for immersion measurement)
F-50 series standard electrode	9611-10D	9096001800	Glass-body electrode (reinforced responsive glass)
Laboratory-use electrode for slurry samples	9677-10D	9096002000	Built-in washable reference electrode (reinforced responsive glass)
Laboratory-use electrode for micro samples	9669-10D	9096001900	Electrode incorporating temperature sensor compatible with micro sample measurement Tip: ϕ3, 55 mm

● pH electrode (without built-in temperature sensor)

Part name	Model	Part number	Remarks
Low-end electrode	6066-10C	9003013400	Glass-body electrode
Electrode for NMR tubes	6069-10C	9003013500	Tip: ϕ3, 180 mm

● pH standard solution

Part name	Model	Part number	Remarks
pH2 standard solution	100-2	9003001500	500 mL Accuracy: ±0.02 pH
pH4 standard solution	100-4	9003001600	
pH7 standard solution	100-7	9003001700	
pH9 standard solution	100-9	9003001800	

Part name	Model	Part number	Remarks
pH2 standard solution powder	150-2	9003002600	Makes 500mL (10 packets) Accuracy: ±0.05 pH
pH4 standard solution powder	150-4	9003002700	
pH7 standard solution powder	150-7	9003002800	
pH9 standard solution powder	150-9	9003002900	

● Internal reference solution

Part name	Model	Part number	Remarks
Internal reference solution	#300	9003003200	250 mL

● Cleaning liquid

Part name	Model	Part number	Remarks
Cleaning liquid for electrodes	#220	9096002500	For pH, ORP, and ION electrodes

5.5.2 Options

Part name			Part number	Remarks
AC adapter for the pH meter	AC adapter		9096003100	Be sure to purchase the cable when purchasing the AC adapter.
	Cable	For Japan	9096003200	
		For US	9096003300	
		For Europe	9096003400	
Soft case			9096005100	
Strap			9096005200	For the meter
Stand for electrode			9096002700	
Stand arm			9096002800	

For any question regarding this product, please contact your local agency, or inquire from the Customer Registration website (www.horiba.co.jp/register)

HORIBA, Ltd.

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ULTRAMETER II™

Operation Manual

MODELS 6Psi & 4P

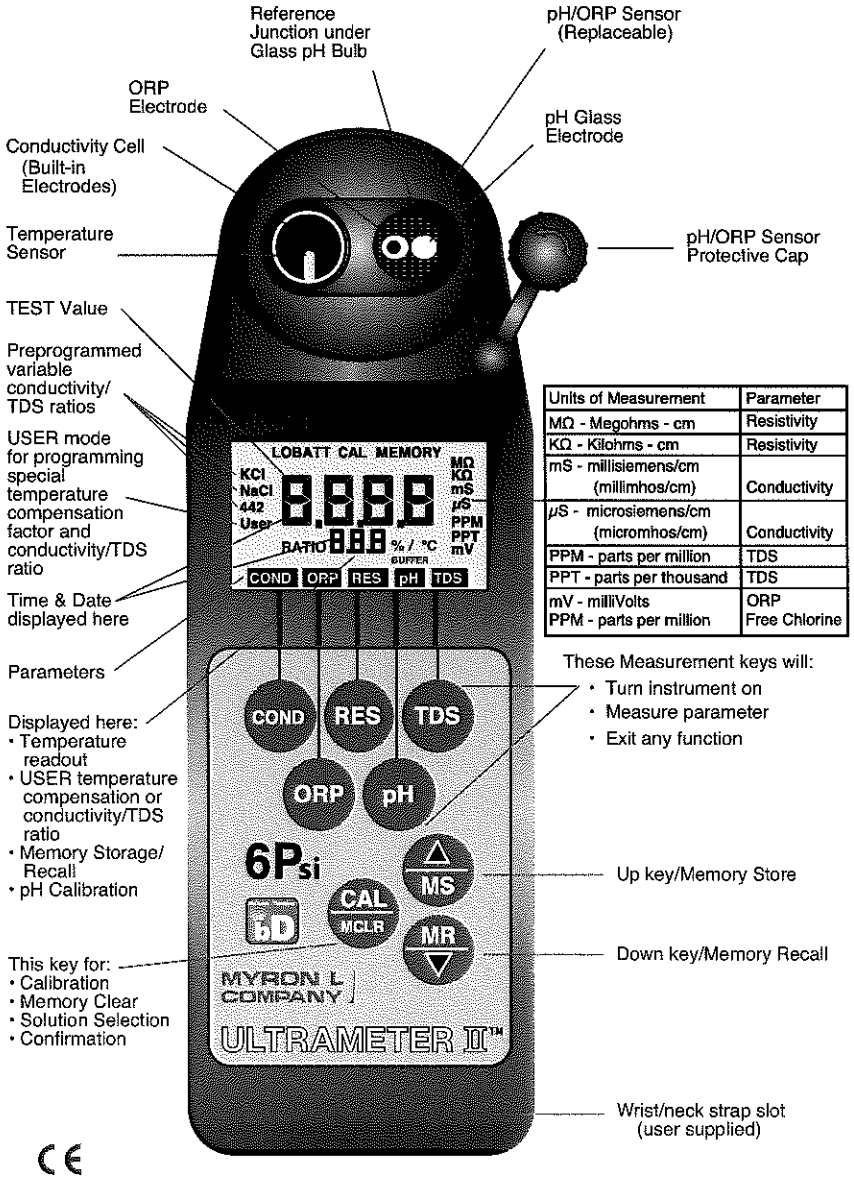
MYRON L COMPANY

Water Quality Instrumentation

Accuracy • Reliability • Simplicity

08 September 10

Instrument Illustration



Units of Measurement	Parameter
MΩ - Megohms - cm	Resistivity
KΩ - Kilohms - cm	Resistivity
mS - millisiemens/cm (millimhos/cm)	Conductivity
µS - microsiemens/cm (micromhos/cm)	Conductivity
PPM - parts per million	TDS
PPT - parts per thousand	TDS
mV - milliVolts PPM - parts per million	ORP Free Chlorine

CE

MODEL 6Psi
Shown with bluDock™ option installed

For detailed explanations see Table of Contents

23mar10

I. INTRODUCTION

Thank you for selecting the feature-packed Ultrameter II™, one of the Myron L Company's latest in an increasing line of instruments utilizing advanced microprocessor-based circuitry and SMT manufacturing processes. This circuitry makes the instrument extremely accurate, reliable and very easy to use.

Model 6Psi has been redesigned to include free chlorine measurements and a LSI & Hardness Calculator for water balance analysis. Both Ultrameter IIs now also feature optional *Bluetooth®* wireless data transfer. Other features include a clock with time and date, memory of up to 100 locations with time and date stamp, the ability of the user to adjust the timeout "Auto OFF", and enhanced performance. See Features and Specifications on pages 2 & 3.

The most exciting new feature is data logging with the ability to download the memory or stored test data wirelessly with its corresponding time, date and unit name. This feature allows the user to create spreadsheets and graphs with ease, and quickly and accurately manipulate data more effectively. The optional bluDock™ and accompanying U2CI software is compatible with most computers using either Microsoft Windows XP, 2000, 2007 or Vista™ or Macintosh OS9.2 or OSX™. The data may be imported into a variety of spreadsheet formats like Microsoft Excel CSV™.
Please Note: Although the Myron L Company has performed extensive testing, we cannot guarantee compatibility of all applications and formats. We suggest testing your application and format for compatibility before relying on it.

For your convenience, a brief set of instructions is provided on the bottom side of your Ultrameter II. A waterproof pocket-sized card with abbreviated instructions is also included with the instrument.

Special note ... Conductivity, resistivity, and TDS require mathematical correction to 25°C values (ref. Temperature Compensation, pg. 37). On the left of the Ultrameter II's liquid crystal display is shown an indicator of the salt solution characteristic used to model temperature compensation of conductivity and its TDS conversion. The indicator may be KCl, NaCl, 442™ or User. Selection affects the temperature correction of conductivity, and the calculation of TDS from compensated conductivity (ref. Conductivity Conversion to Total Dissolved Solids (TDS), pg. 40). The selection can affect the reported conductivity of hot or cold solutions, and will change the reported TDS of a solution. Generally, using KCl for conductivity, NaCl for resistivity, and 442 (Natural Water characteristic) for TDS will reflect present industry practice for standardization. This is how your instrument, as shipped from the factory, is set to operate. For use in sea water desalination for example, both the conductivity and TDS may easily be changed to NaCl.

II. FEATURES and SPECIFICATIONS

A. Features

- *Bluetooth®* wireless download capability with optional bluDock™
- ORP mV to ppm free chlorine conversion (6Psi)
- Langelier Saturation Index & Hardness Calculator (6Psi)
- Superior resolution 4 digit LCD displays full 9999 µS/ppm
- Accuracy of BETTER than ±1% of READING in a handheld instrument ±0.2% at calibration point
- All electrodes are internal for maximum protection
- Improved 4 electrode sensor technology
- Waterproof to 1 meter/3 feet
- Autoranging conductivity/TDS/resistivity
- Prompts for easy pH calibration (6Psi)
- Factory calibrations stored in microprocessor
- 3 conductivity/TDS solution conversions preprogrammed into microprocessor
- "User" mode feature allows:
  Programming your own cond/TDS conversion factor
  Programming your own temperature compensation factor
  Disabling temperature compensation
- Real Time Clock with Time and Date
- Data Logging with TIME and DATE in memory
- Memory stores 100 readings
- User adjustable timeout "Auto OFF"

B. General Specifications

Display	4 Digit LCD
Dimensions (LxWxH)	196 x 68 x 64 mm/ 7.7 x 2.7 x 2.5 in.
Weight	352 g/12.4 oz.
Case Material	VALOX*
Cond/Res/TDS Cell Material	VALOX*
Cond/TDS Electrodes (4)	316 Stainless Steel
Cond/Res/TDS Cell Capacity	5 ml/0.2 oz.
pH/ORP Sensor Well Capacity	1,2 ml (6Psi)/0.04 oz.
Power	9V Alkaline Battery
Battery Life	>100 Hours/5000 Readings
Operating/Storage Temperature	0-55°C/32-132°F
Protection Ratings	IP67/NEMA 6 (waterproof to 1 meter/3 feet)
CE EMI/EMC Ratings (Conformité Européenne)	EN61326-1: 2006 + Annex A: 2008 (hand-held devices) CISPR 11: 2003 IEC 61000-4-2: 2001 and, IEC 61000-4-3: 2002

* ™ SABIC Innovative Plastics IP BV

Additional information is available on our website:
www.myronl.com

MADE IN USA

C. Specification Chart

	pH (6Psi)	ORP (6Psi) Free Chlorine	Conductivity	TDS	Resistivity	Temperature
Ranges	0-14 pH	±999 mV 0.20 - 9.99 ppm	0-9999 µS/cm 10-200 mS/cm in 5 autoranges	0-9999 ppm 10-200 ppt in 5 autoranges	10KΩ - 30MΩ	0-71 °C 32 - 160 °F
Resolution	±.01 pH	±1 mV 0.01 ppm	0.01 (<100 µS) 0.1 (<1000 µS) 1.0 (<10 mS) 0.01 (<100 mS) 0.1 (<200 mS)	0.01 (<100 ppm) 0.1 (<1000 ppm) 1.0 (<10 ppt) 0.01 (<100 ppt) 0.1 (<200 ppt)	0.01 (<100 KΩ) 0.1 (<1000 KΩ) 0.1 (>1 MΩ)	0.1 °C/F
Accuracy	±.01 pH*	±1 mV ± 2.5% of reading ppm**	±1% of reading	±1% of reading	±1% of reading	±0.1 °C
Auto Temperature Compensation	0-71 °C 32-160 °F		0-71 °C 32 - 160 °F	0-71 °C 32 - 160 °F	0-71 °C 32 - 160 °F	
Adjustable Temperature Compensation			0 - 9.99%/ °C	0 - 9.99%/ °C	0 - 9.99%/ °C	
Cond/TDS Ratios Preprogrammed			KCl, NaCl, 442™			
Adjustable Cond/TDS Ratio Factor			0.20 - 7.99			

*± .2 pH in presence of RF fields ≥ 3 V/m and > 300 MHz **Given water sanitized by chlorine only, 5<pH<9, and 20°C<T<30°C

The LSI Calculator (6Psi) hardness range is limited to 0.0 - 1710 ppm and 0.0 - 100 grains of hardness.

D. Warranty/Service

The Myron L Ultrameter II, excluding the pH/ORP sensor (6Psi), has a Two (2) Year Limited Warranty. The pH/ORP sensor (6Psi) has a Six (6) Month Limited Warranty for materials and workmanship. If an instrument fails to operate properly, see Troubleshooting Chart, pg. 34. The battery and pH/ORP sensor are user-replaceable. For other service, return the instrument prepaid to the Myron L Company.

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If, in the opinion of the factory, failure was due to materials or workmanship, repair or replacement will be made without charge. A reasonable service charge will be made for diagnosis or repairs due to normal wear, abuse or tampering. This warranty is limited to the repair or replacement of the Ultrameter II only. The Myron L Company assumes no other responsibility or liability.

E. Ultrameter II Models

ULTRAMETER II MODELS	4P	6Psi
PARAMETERS	Conductivity/TDS Resistivity/Temp.	Conductivity/TDS/pH/Resistivity ORP mV and ppm free chlorine/Temp.

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III. RULES of OPERATION

A. Operation

Using the instrument is simple:

- Individual or multiple parameter readings may be obtained by filling individual sensors or entire cell cup area.
- Rinse the conductivity cell or pH/ORP sensor (6Psi) well with test solution 3 times and refill. Temperature and/or measurement extremes will require additional rinses for maximum accuracy.
- Press the desired measurement key to start measurement. Pressing the key again restarts the 15 second auto "off" timer.
- Note the value displayed or press the MS key to store the reading (ref. Memory Storage, pg. 21). It's that simple!

B. Characteristics of the Keys

- Though your Ultrameter II has a variety of sophisticated options, it is designed to provide quick, easy, accurate measurements by simply pressing one key.
- All functions are performed one key at a time.
- There is no "off" key. After 15 seconds of inactivity the instrument turns itself off (60 seconds in CAL mode). User adjustable up to 75 seconds.
- Rarely is it necessary to press and *hold* a key (as in Procedure to Select a Solution, pg. 11; or Conductivity or TDS Calibration, pg. 15).

C. Operation of the Keys (See Instrument Illustration, pg. i)

1. Measurement Keys in General

Any of the 5 measurement keys in the upper part of the keypad turns on the instrument in the mode selected. The mode is shown at the bottom of the display, and the measurement units appear at the right. Pressing a measurement key does this even if you are in a calibration sequence and also serves to cancel a change (ref. Leaving Calibration, pg. 14).

2. COND, RES and TDS Keys

These 3 keys are used with solution in the Conductivity Cell.
Precautions:

- While filling cell cup ensure no air bubbles cling on the cell wall.
- If the proper solution is not selected (KCl, NaCl, 442 or User), refer to Why Solution Selection is Available, pg. 11 and Procedure to Select a Solution, pg. 11.

a. COND Key

Solution to be tested is introduced into the conductivity cell and a press of (COND) displays conductivity with units on the right. On the left is shown the solution type selected for conductivity.

b. RES Key

A press of (RES) displays resistivity with units on the right. On the left is shown solution type selected for resistivity (ref. Solution Selection, pg. 11). The range of display of resistivity is limited to between 10 kilohms (KΩ) and 30 megohms (MΩ). A solution outside that range will only show [- - - -] in the display.

c. TDS Key

A press of (TDS) displays Total Dissolved Solids with units on the right. This is a display of the concentration of material calculated from compensated conductivity using the characteristics of a known material. On the left is shown solution type selected for TDS (ref. Solution Selection, pg. 11).

3. pH and ORP Keys (6Psi)

Measurements are made on solution held in the pH/ORP sensor well (ref. pH and ORP, pg. 43). The protective cap is removed and the sensor well is filled and rinsed with the sample enough times to completely replace the storage solution.

After use, the pH/ORP sensor well must be refilled with Myron L Storage Solution, and the protective cap reinstalled securely (ref. Maintenance of the pH/ORP Sensor, pg. 9 and Cleaning Sensors, 2. pH/ORP, pg. 32).

a. pH Key (6Psi)

A press of (pH) displays pH readings. No units are displayed on the right.

b. ORP Key (6Psi)

A press of (ORP) displays Oxidation-Reduction Potential/REDOX reading in millivolts; "mV" is displayed.

NOTE: See New ORP ppm Free Chlorine Feature, pg. 48, to change units to ppm free chlorine.

4. CAL/MCLR Key

A press of (CAL/MCLR) allows you to enter the calibration mode while measuring conductivity, TDS or pH. Once in CAL mode, a press of this key accepts the new value. If no more calibration options follow, the instrument returns to measuring (ref. Leaving Calibration, pg. 14).

If (CAL/MCLR) is held down for about 3 seconds, "CAL" mode is not entered, but "**SEL**" appears to allow Solution Selection (ref. pg. 11) with the Up or Down keys. As in calibration, the CAL key is now an "accept" key.

While reviewing stored records, the MCLR side of the key is active to allow clearing records (ref. Clearing a Record/Memory Clear, pg. 21).

5. UP or DOWN Keys

While measuring in any parameter, the (▲ MS) or (MR ▼) keys activate the Memory Store and Memory Recall functions.
While in "CAL" mode, the keys step or scroll the displayed value up or down. A single press steps the display and holding either key scrolls the value rapidly.
While in Memory Recall, the keys scroll the display up and down through the stack of records (ref. Memory Recall, pg. 21).

IV. AFTER USING THE ULTRAMETER II

A. Maintenance of the Conductivity Cell

Rinse out the cell cup with clean water. Do not scrub the cell. For oily films, squirt in a foaming non-abrasive cleaner and rinse (ref. Cleaning Sensors, pg. 32). Even if a very active chemical discolors the electrodes, this does not affect the accuracy; leave it alone.

B. Maintenance of the pH/ORP Sensor (6Psi)

The sensor well must be kept wet with a saline solution. Before replacing the rubber cap, rinse and fill the sensor well with Myron L pH Sensor Storage Solution. If unavailable, use an almost saturated KCl solution, pH 4 buffer or a saturated solution of table salt and tap water (ref. pH and ORP Practices to Maintain Calibration, pg. 20). NEVER USE DISTILLED WATER.

V. SPECIFIC RECOMMENDED MEASURING PROCEDURES

If the proper solution is not selected (KCl, NaCl, 442 or User), see Solution Selection, pg. 11.

NOTE: *After sampling high concentration solutions or temperature extremes, more rinsing may be required. When sampling low conductivity solutions, be sure the pH cap is well seated so that no solution washes into the conductivity cell from around the pH cap.*

A. Measuring Conductivity & Total Dissolved Solids (TDS)

1. Rinse cell cup 3 times with sample to be measured. (This conditions the temperature compensation network and prepares the cell.)

2. Refill cell cup with sample.

3. Press (COND) or (TDS).

4. Take reading. A display of [- - - -] indicates an overrange condition.

B. Measuring Resistivity

Resistivity is for low conductivity solutions. In a cell cup the value may drift from trace contaminants or absorption from atmospheric gasses, so measuring a flowing sample is recommended.

1. Ensure pH protective cap is secure to avoid contamination.
2. Hold instrument at 30° angle (cup sloping downward).
3. Let sample flow continuously into conductivity cell with no aeration.
4. Press (RES) key; use best reading.

NOTE: *If reading is lower than 10 kilohms display will be dashes: [- - - -]. Use Conductivity.*

C. Measuring pH (6Psi)

1. Remove protective cap by rotating while grasping and pulling up.
2. Rinse pH/ORP sensor well and conductivity cell cup 3 times with sample to be measured. Shake out each sample to remove any residual liquid.
3. Refill both sensor well and cell cup with sample.
4. Press (pH).
5. Note value displayed.
6. **IMPORTANT:** After use, fill pH/ORP sensor well with Myron L pH Sensor Storage Solution and replace protective cap. If Myron L pH Sensor Storage Solution is unavailable, use a strong KCl solution, a pH 4 buffer, or a saturated solution of table salt and tap water (ref. Cleaning Sensors, 2. pH/ORP, pg. 32). *Do not allow pH/ORP sensor to dry out.*

D. Measuring ORP (6Psi)

1. Remove protective cap by rotating while grasping and pulling up.
2. Rinse sensor well and cell cup 3 times with sample to be measured. Shake out each sample to remove any residual liquid.
3. Refill both sensor well and cell cup with sample.
4. Press (ORP).
5. Take reading.

NOTE: When ppm free chlorine units are selected (ref. free chlorine feature, pg. 48), annunciators alert you when the concentration is outside the specified measurement range. "**Or**" (over range) will display when the

concentration is over the range limit (> 9.99 ppm). "**Ur**" (under range) will display when the concentration is below the range limit (< 0.20 ppm).

6. **IMPORTANT:** After use, fill pH/ORP sensor well with Myron L pH Sensor Storage Solution and replace protective cap. If Myron L pH Sensor Storage Solution is unavailable, use a strong KCl solution, a pH 4 buffer, or a saturated solution of table salt and tap water (ref. Cleaning Sensors, 2. pH/ORP, pg. 32). *Do not allow pH/ORP sensor to dry out.*

NOTE: FOR INFORMATION REGARDING THE ORP mV TO ppm CONVERSION FEATURE, PLEASE SEE SECTION XXVI. ADDENDUM, PG. 48.

VI. SOLUTION SELECTION

A. Why Solution Selection is Available

Conductivity, resistivity, and TDS require temperature correction to 25°C values (ref. Standardized to 25°C, pg. 37). Selection determines the temperature correction of conductivity and calculation of TDS from compensated conductivity (ref. Cond. Conversion to TDS, pg. 40).

B. The 4 Solution Types

On the left side of the display is the salt solution characteristic used to model temperature compensation of conductivity and its TDS conversion. Generally, using KCl for conductivity, NaCl for resistivity, and 442 (Natural Water characteristic) for TDS will reflect present industry practice for standardization. This is how your instrument is shipped from the factory (ref. Solution Characteristics, pg. 40).

The User selection allows a custom value to be entered for the temperature compensation of conductivity and also the conversion ratio if measuring TDS.

C. Calibration of Each Solution Type

There is a separate calibration for each of the 4 solution types. Note that calibration of a 442 solution does not affect the calibration of a NaCl solution. For example: Calibration (ref. Conductivity or TDS Calibration, pg. 15) is performed separately for each type of solution one wishes to measure (ref. Conductivity/TDS Standard Solutions, pg. 36).

D. Procedure to Select a Solution

NOTE: Check display to see if solution displayed (KCl, NaCl, 442 or User) is already the type desired. If not:

1. Press (COND), (RES) or (TDS) to select the parameter on which

you wish to change the solution type.

2. Press and hold (CAL/MCLR) key for 3 seconds to make "**SEL**" appear (see Figure 1). For demonstration purposes, all 4 solution types are shown simultaneously.

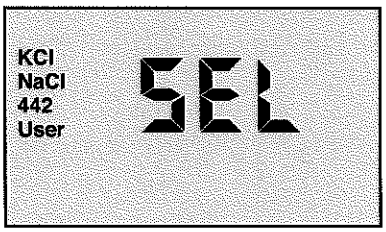


Figure 1

3. Use the (▲/MS) or (MR/▼) key to select type of solution desired (ref. Solution Characteristics, pg. 40). The selected solution type will be displayed: KCl, NaCl, 442 or User.

4. Press (CAL/MCLR) to accept new solution type.

E. Application of User Solution Type

1. User Programmable Temperature Compensation (Tempco)

This feature allows you to change your Ultrameter II's temperature compensating factor to another factor between 0-9.99%/°C (ref. Temperature Compensation, pg. 37). This feature does not apply to pH or ORP.

a. As in Procedure to Select a Solution, pg. 11, select "User" mode.

b. With "User" mode now selected, press (CAL/MCLR). You may now adjust a temperature compensation from .00%/°C to 9.99%/°C, by pressing (▲/MS) or (MR/▼). See example in Figure 2.

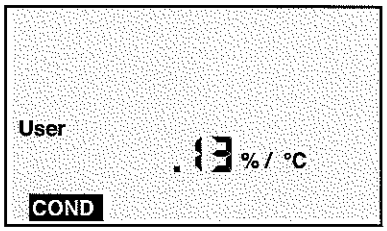


Figure 2

c. Press (CAL/MCLR) twice to skip calibration adjustment and accept the new tempco (3 times if in TDS mode). You are now ready to measure samples with your new temperature compensation factor.

2. Disabling Temperature Compensation

a. Select "User" mode (ref. Procedure to Select a Solution, pg. 11).

b. With "**User**" selected, press (CAL/MCLR). If the display does not show .00%/°C, hold (MR ▼) long enough to bring the tempco to .00%/°C (see Figure 3).

c. Press (CAL/MCLR) twice (3 times if in TDS mode). Temperature compensation is now disabled (=0) for measurements in "User" mode.

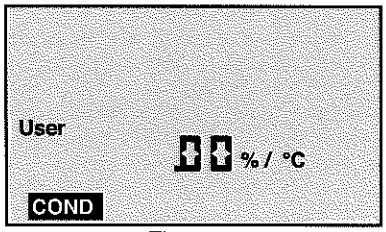


Figure 3

3. User Programmable Conductivity to TDS Ratio

This feature allows you to select a custom conductivity to TDS conversion ratio within the range of 0.20-7.99 for "User" mode measurements.
To determine the conversion ratio for a custom solution of **known** TDS ppm value, measure the solution conductivity **at 25°C** with the Ultrameter II and divide the ppm value by the μS value. For example, a solution of known 75 ppm TDS and measured 100 μS conductivity at 25°C would have a conversion ratio of 75/100 or 0.75. Enter the new conversion ratio as follows:

a. While in "User" mode, press (TDS).

b. Press (CAL/MCLR) twice (to skip over tempco adjustment) and "**RATIO**" will appear (see Figure 4).

c. Adjust with (▲ MS) or (MR ▼) key until new conversion ratio is displayed.

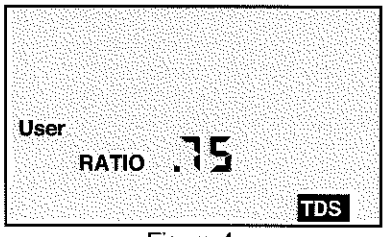


Figure 4

d. Press (CAL/MCLR) twice (to skip over calibration adjustment) to accept new conversion ratio. You are now ready to measure samples with the new conductivity/TDS ratio.

In these first six sections, you have learned all you need to make accurate measurements. The following sections contain calibration, advanced operations and technical information.

VII. CALIBRATION

A. Calibration Intervals

Generally, calibration is recommended about once per month with Conductivity or TDS solutions. Calibration with pH solutions should be checked twice a month. Calibration of ORP is not necessary (ref. CALIBRATION INTERVALS, pg. 19).

B. Rules for Calibration of the Ultrameter II

1. Calibration Steps

a. Starting Calibration

Calibration is begun by pressing (CAL/MCLR) while measuring Conductivity, TDS or pH. Measuring continues, but the "**CAL**" icon is on, indicating calibration is now changeable.

The reading is changed with the (▲/MS) and (MR/▼) keys to match the known value. The calibration for each of the 4 solution types may be performed in either conductivity or TDS mode.

Depending on what is being calibrated, there may be 1, 2 or 3 steps to the calibration procedures.

	KCl, NaCl or 442	User
Cond	Gain only	Tempco, then Gain
Res	Done in conductivity	Done in conductivity or TDS
TDS	Gain only	Tempco, Ratio, then Gain
pH	7, acid and/or base (6Psi)	
ORP	Zero set with pH 7 automatically (6Psi)	

Once in "CAL" mode, the (CAL/MCLR) key becomes an "ACCEPT" key. At each point, pressing (CAL/MCLR) accepts the new calibration value and steps you to the next adjustment (or out of "CAL" mode if there are no more steps).

To bypass a calibration step, simply press (CAL/MCLR) to accept the present value as is.

b. Leaving Calibration

Calibration is complete when the "**CAL**" icon goes out. Pressing any

measurement key cancels changes not yet accepted and exits calibration mode.
Leaving pH after the 2nd buffer results in the same gain being entered in place of the 3rd buffer.

2. Calibration Limits

There are calibration limits. A nominal "FAC" value is an ideal value stored by the factory. Attempts to calibrate too far, up or down, from there will cause the displayed value to be replaced with "FAC". If you accept it (press the "Cal" key), you will have the original default factory calibration for this measurement. The need to calibrate so far out that "FAC" appears indicates a procedural problem, incorrect standard solution, a very dirty cell cup or an aging pH/ORP sensor (ref. Troubleshooting Chart, pg. 34).

C. Calibration Procedures

1. Conductivity or TDS Calibration

a. Rinse conductivity cell three times with proper standard (KCl, NaCl, or 442) (ref. Cond/TDS Standard Solutions, pg. 36). For user calibration see User Calibration Conductivity/TDS below.

b. Refill conductivity cell with same standard. KCl-7000 shown.

c. Press (COND) or (TDS), then press (CAL/MCLR); "**CAL**" icon will appear on the display (see Figure 5).

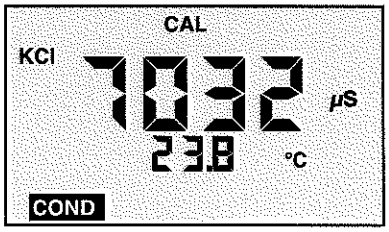


Figure 5

d. Press (▲/MS) or (MR/▼) to step the displayed value toward the standard's value (7032 > 7000) or hold a key down to scroll rapidly through the reading.

e. Press (CAL/MCLR) once to confirm new value and end the calibration sequence for this particular solution type. If another solution type is also to be measured, change solution type now and repeat this procedure.

2. User Calibration Conductivity/TDS

Instrument must be in "User" mode, see Solution Selection, pg. 11.

a. Rinse conductivity cell three times with your standard.

b. Refill conductivity cell with same standard.

c. Press COND or TDS, then press CAL/MCLR twice in COND/three times in TDS. The "**CAL**" icon will appear on the display.

d. Press ▲/MS or MR/▼ to step the displayed value toward the standard's value or hold a key down to scroll rapidly through the reading.

e. Press CAL/MCLR once to confirm new value and end the calibration sequence for this particular solution type.

3. Resistivity Calibration

Resistivity is the reciprocal of Conductivity. To calibrate resistivity, calibrate conductivity for the solution type you wish to measure (ref. Conductivity or TDS Calibration, pg. 15).

4. Reloading Factory Calibration (Cond or TDS)

If calibration is suspect or known to be incorrect, and no standard solution is available, the calibration value can be replaced with the original factory value for that solution. This "FAC" value is the same for all Ultrameter IIs, and returns you to a known state without solution in the cell. The "FAC" internal electronics calibration (which bypasses the electrodes and cell) is not intended to replace calibration with conductivity/TDS standard solutions. If another solution type requires resetting, change solution type and repeat this procedure.

a. Press COND or TDS.

b. Press CAL/MCLR. (If in "User" solution mode, press CAL key twice if in Conductivity, and three times if in TDS to skip over tempco and ratio adjustments.)

c. Press ▲/MS key until "**FAC**" appears and release.

d. Press CAL/MCLR to accept the factory calibration setting.

5. pH Calibration (6Psi)

Important: Always "zero" your Ultrameter II with a pH 7 buffer solution before adjusting the gain with acid or base buffers, i.e., 4 and/or 10, etc.

a. pH Zero Calibration (6Psi)

1. Rinse sensor well and cell cup 3 times with 7 buffer solution.

2. Refill both sensor well and cell cup with 7 buffer solution.

3. Press (pH) to verify the pH calibration. If the display shows 7.00, skip the pH Zero Calibration and proceed to section b. pH Gain Calibration.

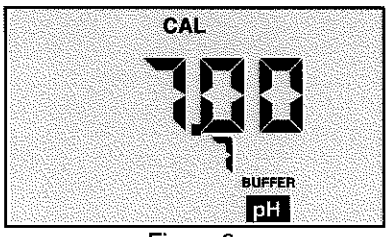


Figure 6

4. Press (CAL/MCLR) to enter calibration mode. The "**CAL**", "**BUFFER**" and "**7**" annunciators will appear (see Figure 6). Displayed value will be the uncalibrated sensor.

NOTES: *If a wrong buffer is added (outside of 6-8 pH), "**7**" and "**BUFFER**" will flash, and the Ultrameter II will not adjust.*
The uncalibrated pH value displayed in step 4 will assist in determining the accuracy of the pH sensor. If the pH reading is above 8 with pH 7 buffer solution, the sensor well needs additional rinsing or the pH sensor is defective and needs to be replaced.

5. Press (▲/MS) or (MR/▼) until the display reads 7.00.

NOTE: *Attempted calibration of >1 pH point from factory calibration will cause "**FAC**" to appear. This indicates the need for sensor replacement (ref. Troubleshooting pg. 34) or fresh buffer solution. The "FAC" internal electronic calibration is not intended to replace calibration with pH buffers. It assumes an ideal pH sensor. Each "FAC" indicates a factory setting for that calibration step (i.e., 7, acid, base).*

You may press (CAL/MCLR) to accept the preset factory value, or you may

reduce your variation from factory setting by pressing (▲ MS) *or* (MR ▼).

6. Press (CAL MCLR) to accept the new value. The pH Zero Calibration is now complete. You may continue with pH Gain Calibration or exit by pressing any measurement key.

b. pH Gain Calibration (6Psi)

Important: Always calibrate or verify your Ultrameter II with a pH 7 buffer solution before adjusting the gain with acid or base buffers, i.e., 4 and/or 10, etc. Either acid or base solution can be used for the 2nd point "Gain" calibration and then the opposite for the 3rd point. The display will verify that a buffer is in the sensor well by displaying either "**Acd**" or "**bAS**".

1. The pH calibration mode is initiated by either completion of the pH Zero Calibration, or verifying 7 buffer and pressing the (CAL MCLR) key twice while in pH measurement mode.

2. At this point the "**CAL**", "**BUFFER**" and "**Acd**" or "**bAS**" annunciators will be displayed (see Figures 7 and 8).

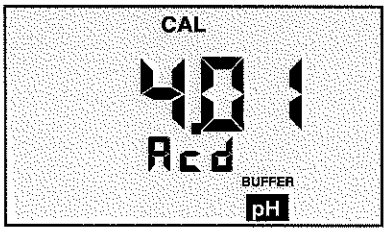


Figure 7

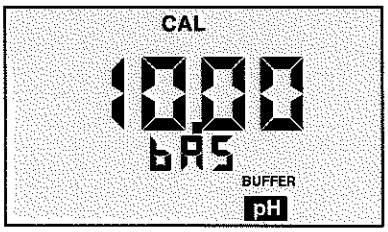


Figure 8

NOTE: *If the "**Acd**" and "**bAS**" indicators are blinking, the unit is indicating an error and needs either an acid or base solution present in the sensor well.*

3. Rinse sensor well 3 times with acid or base buffer solution.

4. Refill sensor well again with same buffer solution.

5. Press (▲ MS) or (MR ▼) until display agrees with buffer value.

6. Press (CAL/MCLR) to accept 2nd point of calibration. Now the display indicates the next type of buffer to be used.

Single point Gain Calibration is complete. You may continue for the 3rd point of Calibration (2nd Gain) or exit by pressing any measurement key. Exiting causes the value accepted for the buffer to be used for both acid and base measurements.
To continue with 3rd point calibration, use basic buffer if acidic buffer was used in the 2nd point, or vice-versa. Again, match the display to the known buffer value as in step 2 and continue with the following steps:

7. Repeat steps 3 through 6 using opposite buffer solution.

8. Press (CAL/MCLR) to accept 3rd point of calibration, which completes the Calibration procedure. Fill sensor well with Myron L Storage Solution and replace protective cap.

6. ORP Calibration (6Psi)

ORP electrodes rarely give false readings without problems in the reference electrode. For this reason, and because calibration solutions for ORP are highly reactive and potentially hazardous, your Ultrameter II has an electronic ORP calibration. This causes the zero point on the reference electrode to be set whenever pH 7 calibration is done.

7. Temperature Calibration

Temperature calibration is not necessary in the Ultrameter II.

VIII. CALIBRATION INTERVALS

There is no simple answer as to how often one should calibrate an instrument. The Ultrameter II is designed to not require frequent recalibration. The most common sources of error were eliminated in the design, and there are no mechanical adjustments. Still, to ensure specified accuracy, any instrument must be checked against chemical standards occasionally.

A. Suggested Intervals

On the average, we expect calibration need only be checked monthly for the Conductivity, RES or TDS functions. The pH (6Psi) function should be checked every 2 weeks to ensure accuracy. Measuring some solutions will require more frequent intervals.

B. Calibration Tracking Records

To minimize your calibration effort, keep records. If adjustments you are making are minimal for your application, you can check less often. Changes in conductivity calibration should be recorded in percent. Changes in pH calibration (6Psi) are best recorded in pH units.

Calibration is purposely limited in the Ultrameter II to ±10% for the conductivity cell, as any change beyond that indicates damage, not drift. Likewise, calibration changes are limited to ±1 pH unit (6Psi), as any change beyond that indicates the end of the sensor's lifetime and replacement is recommended.

C. Conductivity, RES, TDS Practices to Maintain Calibration

1. Clean oily films or organic material from the cell electrodes with foaming cleaner or mild acid. Do not scrub inside the cell.

2. Calibrate with solutions close to the measurements you make. Readings are compensated for temperature based on the type of solution. If you choose to measure tap water with a KCl compensation, which is often done (ref. An Example of 2 different solution selections and the resulting compensation, pg. 38), and you calibrate with 442 solution because it is handy, the further away from 25°C you are, the more error you have. Your records of calibration changes will reflect temperature changes more than the instrument's accuracy.

3. Rinse out the cell with pure water after taking measurements. Allowing slow dissolving crystals to form in the cell contaminates future samples.

4. For maximum accuracy, keep the pH sensor cap on tight so that no fluid washes into the conductivity cell.

D. pH and ORP Practices to Maintain Calibration (6Psi)

1. Keep the sensor wet with Myron L Storage Solution.

2. Rinse away caustic solutions immediately after use.

ORP calibration solutions are caustic, and ±5% is considered very accurate. By using the pH zero setting (0 mV = 7 pH) for ORP and precision electronics for detection, the Ultrameter II delivers better accuracy without calibration than a simpler instrument could using calibration solutions.

IX. MEMORY

This feature allows up to 100 readings with their temperatures to be stored simultaneously for later recall. At the same time, the TIME and DATE are also recorded. To download the memory to a computer, (ref. bluDock™ WIRELESS DATA TRANSFER INSTRUCTIONS, pg. 30).

A. Memory Storage

1. While displaying a measurement, press ▲/MS to record the displayed value.

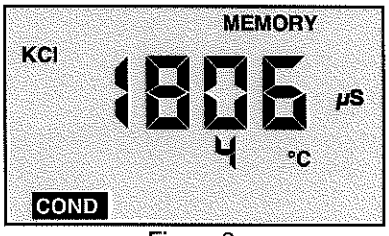


Figure 9

2. "**MEMORY**" will appear and the temperature display will be momentarily replaced by a number (1-100) showing the position of the record. Figure 9 shows a reading of 1806 μS stored in memory record #4.

B. Memory Recall

1. Press any measurement key.
2. Press MR/▼, "**MEMORY**" will appear, and the display will show the last record stored.
3. Press ▲/MS or MR/▼ to scroll to the record location desired (the temperature display alternates between temperature recorded and location number).
4. Press CAL/MCLR to display time and date stamp.
5. Press any measurement key to leave memory recall or allow to automatically turn off.

C. Clearing a Record/Memory Clear

After recalling a certain record location, press and HOLD CAL/MCLR to clear that memory. This space will be the place for the next memory record, unless you scroll to another empty position before ending the recall sequence. The next memory stored will go into the next highest available memory location.

Example: You have locations 1-7 filled and wish to clear the conductivity reading stored in record location #**3** and replace it with a pH reading.

1. Press (MR ▼) and scroll to location #**3**.
2. Press and HOLD (CAL MCLR) to clear old record #**3**.
3. Fill pH/ORP sensor well with sample.
4. Press (pH) to measure sample and press (▲ MS) to store reading in location #**3**.
5. The next memory stored will go into location #**8**.
6. To clear **all** records: After pressing (MR ▼), scroll down. "**CLr ALL**" will be displayed (see Figure 10).
7. Press (CAL MCLR). All records will be cleared.

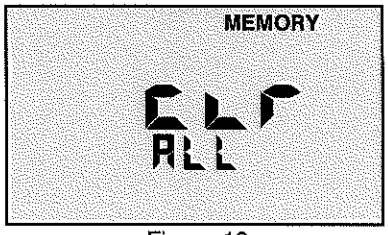


Figure 10

X. TIME and DATE

The Time and Date may easily be changed as you travel.

A. Setting TIME

Time is always displayed in 24 hour time.
Example shown in Figure 11, 16:05 equals 4:05 PM.

1. Press (COND).
2. Press (MR ▼) until the time is displayed (scrolling through stored readings, PC OFF, and CLr ALL to time, e.g., "**16:05**").
3. Press (CAL MCLR) to initiate. "**CAL**" will be displayed along with the time (see Figure 11).

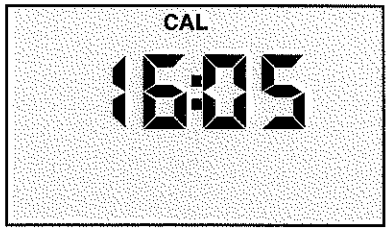


Figure 11

4. Press the (▲ MS) or (MR ▼) to change the time.

5. Press (CAL MCLR) to accept the change (new time).

B. Setting DATE

Example shown in Figure 12 is in US format, i.e., mo/dy/yr. **NOTE:** The default format is US. Date format may be changed (ref. Date Format "US and International (Int)", pg. 24).

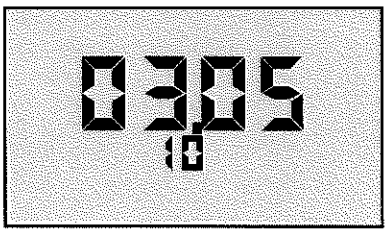


Figure 12

1. Press (COND).

2. Press (MR ▼) repeatedly until the date is displayed (scrolling through stored readings, PC OFF, CLr ALL and time to the date, e.g., **03.05 10** (Figure 12), March 5, 2010).

3. Press (CAL MCLR) to initiate. "**CAL**" will be displayed along with the YEAR (see Figure 13).

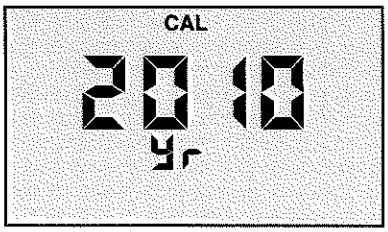


Figure 13

4. Press (▲ MS) or (MR ▼) to change the YEAR.

5. Press (CAL MCLR) to accept the change (new year).

6. Press (▲ MS) or (MR ▼) to change the month.

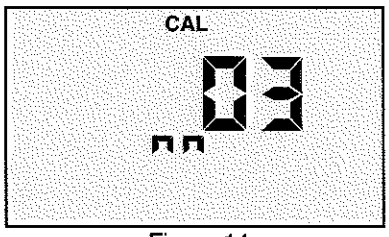


Figure 14

7. Press (CAL MCLR) to accept the change (new month), (see Figure 14).

8. Press the (▲ MS) or (MR ▼) to change the day.

9. Press (CAL MCLR) to accept the change (new day) (see Figure 15).

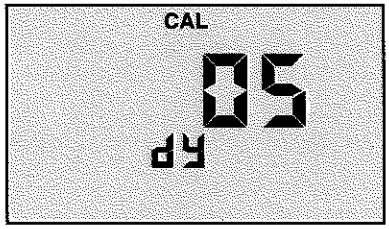


Figure 15

C. DATE FORMAT "US & International (Int)"

1. Press (COND).

2. Press (MR ▼) repeatedly until the format is displayed (scrolling through stored readings, PC OFF, CLr ALL, time and date to date **format**).

3. Press (CAL MCLR) to change. Display will now indicate other format (see Figures 16 & 17).

4. Press any measurement key or allow to automatically turn off.

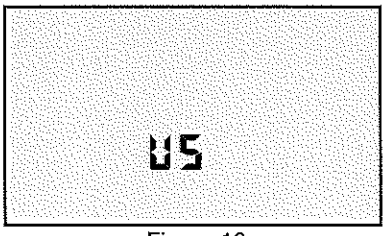


Figure 16

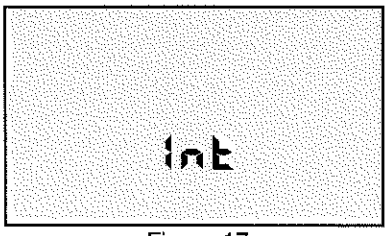


Figure 17

XI. TEMPERATURE FORMAT "Centigrade & Fahrenheit"

1. Press (COND).

2. Press (MR ▼) to display the stored memory records.

3. Press (MR ▼) repeatedly until you pass the "**US**" or "**Int**" date

format location. The display will show a "**C**" or "**F**" (see Figures 18 and 19).

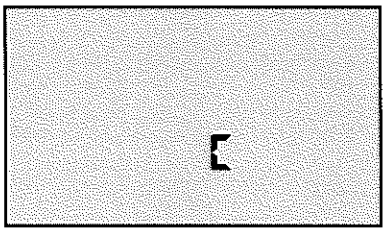


Figure 18

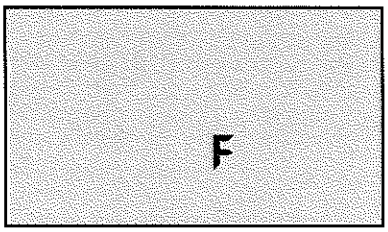


Figure 19

4. Press (CAL/MCLR) to switch units.

5. Press (COND) to accept unit preference for all temperature readings.

NOTE: *Tempco will still be shown in %/°C.*

XII. TOTAL RETURN to FACTORY SETTINGS "FAC SEL"

There may come a time when it would be desirable to quickly reset all the recorded calibration values in the instrument back to the factory settings. This might be to ensure all calibrations are set to a known value, or to give the instrument to someone else free of adjustments or recorded data for a particular application.
NOTE: All stored data will be lost.

1. Press (COND).

2. Press (MR ▼) to display the stored memory records.

3. Press (MR ▼) repeatedly until you pass the CLr ALL and the C-F locations. The display will show a "**FAC SEL**" (see Figure 20).

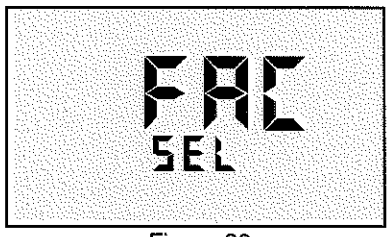


Figure 20

4. Press (CAL/MCLR) to accept the resetting. Display will return to Conductivity.

XIII. CELL CHECK

The cell check verifies the cleanliness of the conductivity/TDS/resistivity sensor. In normal use the cell may become dirty or coated and require cleaning. If the display is showing "**.00**" when the cell cup is dry, the sensor is probably clean. However, when testing high purity water in resistivity ("RES") mode improved accuracy may be desired. No matter what a manufacturer claims, a sensor can and will become contaminated or coated and, therefore, require cleaning. A true 4-wire sensor, as in the Ultrameter II, helps to mitigate contamination, but NO SENSOR IS 100% IMMUNE.

1. Press (COND).

2. Press (MR ▼) to display the stored memory records.

3. Press (MR ▼) repeatedly until you pass the FAC SEL location. The display will show a "**CELL ch**" (see Figure 21).

4. Press (CAL MCLR) to test.

   If cell is clean, "**Good**" will momentarily be displayed (see Figure 22). If cell is dirty, "**CELL cLn**" will be displayed (see Figure 23) (ref. Cleaning Sensors, pg. 32).

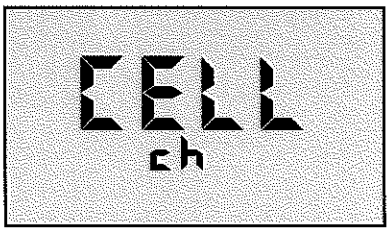


Figure 21

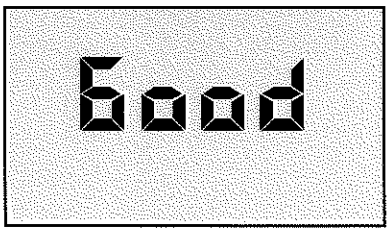


Figure 22

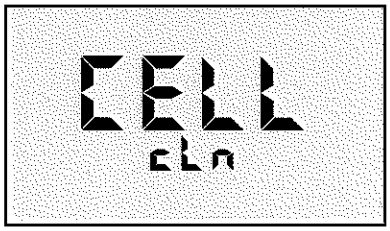


Figure 23

XIV. AUTO OFF

Auto off allows the user to adjust the time the instrument is ON (up to 75 seconds) after each press of a key. Default time is 15 seconds with 60 seconds in "CAL" (calibration) mode.

1. Press (COND).

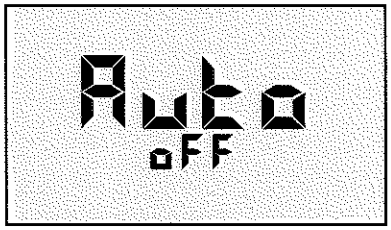


Figure 24

2. Press (MR ▼) to display the stored memory records.

3. Press (MR ▼) repeatedly until you pass the CELL ch location.

The display will show "**Auto oFF**" (see Figure 24).

4. Press (CAL MCLR) to initiate. "**CAL**" will be displayed along with "**15 SEC**" or current Auto oFF value (see Figure 25).

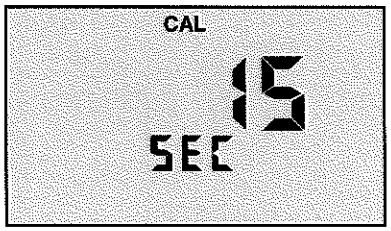


Figure 25

5. Press (▲ MS) or (MR ▼) to change the amount of time (see Figure 26). Maximum time is shown.

6. Press (CAL MCLR) to accept the change (new time).

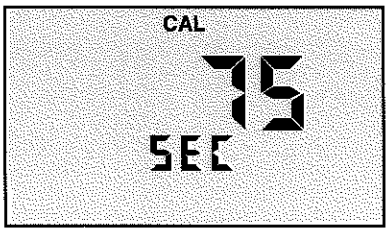


Figure 26

XV. "USER" MODE CALIBRATION LINC™ FUNCTION

The Linc™ function allows easy calibration when in "User" mode and the user does not have a user standard solution to calibrate the instrument. This function will ensure more repeatable and accurate measurements than many other calibration methods. It is recommended that this function be used to provide the highest degree of confidence when the Ultrameter II is used in "User" mode. When Linc is used, the "User" mode is linked to another standard, i.e., if User and KCl are linked, a KCl standard solution is used to calibrate the instrument. It is that simple.

A. Calibration of Ultrameter II for use in "User" mode

1. Press (COND) or (TDS) key.

2. Calibrate the unit using a Standard Solution (ref. CALIBRATION, pg. 14).

3. Place the Ultrameter II in "User" mode (ref. SOLUTION SELECTION, pg. 11).

4. Verify/Set the calibration linc. (See below – Setting "User" Mode Calibration "Linc".)

B. Setting "User" mode Calibration "Linc"

The Linc function sets or "links" the calibration gain factor of a Standard Solution to the User solution mode. Once set, the "Linc" will stay intact with future calibrations unless the Linc has been canceled. For more information on canceling the "User" mode Calibration Linc refer to the section "Canceling "User" mode Calibration "Linc"", pg. 29.
Follow the steps below to set either the KCl, NaCl or 442 calibration factor to the User solution mode.

1. Press measurement key desired to be "Linked", i.e., (COND), (RES) or (TDS).

2. Place the Ultrameter II in "User" mode (ref. SOLUTION SELECTION, pg. 11, for selecting the "User" mode).

3. Press (MR ▼) arrow key until the menu "**Linc**" appears (see Figure 27).

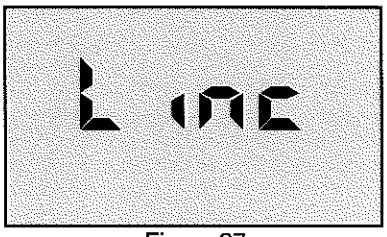


Figure 27

4. Press (CAL MCLR) key. The instrument will display "**SEL**" and the "**User**" Icon (see Figure 28).

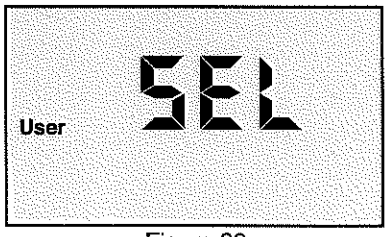


Figure 28

Any additional display of KCl, NaCl or 442 icons indicates a "Linc" between the User solution and the other solution displayed.

5. Press (▲ MS) or (MR ▼) keys to select a Standard Solution to be linked to the "User" mode calibration constant (see Figure 29). "**User**" is linked to "**KCl**".

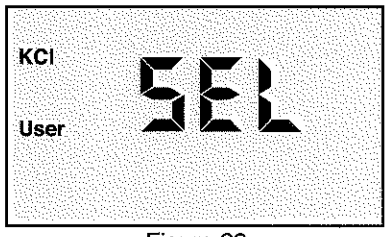


Figure 29

If none of the Solution Selection icons are displayed (i.e., KCl, NaCl or 442), nothing has been linked to "User" mode.

6. Press (CAL/MCLR) key to accept the setting. Pressing any of the measurement keys will exit without changing the setting. "User" mode "Linc" is now complete. The "User" mode will now use the calibration gain constant used for the calibration of the Standard Solution as outlined above.

C. Canceling "User" Mode Calibration "Linc"

The Ultrameter II must be in "User" linked mode in order to cancel the "Linc" (ref. SOLUTION SELECTION, pg. 11).

1. Press "Linked" measurement key (COND), (RES) or (TDS).

   Two solution icons will be shown in the left side of display - "**User**" and another, e.g., "**KCl**".

2. Press (MR ▼) key until the menu "**Linc**" appears (see Figure 27).

3. Press (CAL/MCLR) key; the instrument will display both "**SEL**" and the "**User**" Icon.

4. Press (MR ▼) key until "**User**" is the only solution icon being displayed.

5. Press (CAL/MCLR) key.

6. The "User" mode calibration "Linc" has now been canceled.

NOTES:

1. To maintain repeatability, use the same standard solutions for future calibrations.

2. Calibration of the Ultrameter II Gain Factor for "User" mode is not available when the calibration linc has been established. The other calibration functions (i.e., Temperature Compensation %/C settings and TDS Ratio settings) are still intact. To perform a calibration of the "User" mode as described in User Calibration Conductivity/TDS, pg. 15, the "User" mode Linc should be canceled. See above, "Canceling "User" mode calibration "Linc"".

3. Once a "Linc" has been established for "User" mode , the "Linc" will apply to all measurement modes using User solution selection (i.e., TDS/User, Cond/User or Res/User).

XVI. bluDock™ WIRELESS DATA TRANSFER INSTRUCTIONS

NOTE: *Bluetooth®* is a registered trademark of Bluetooth SIG. The bluDock Bluetooth module is a registered Bluetooth device.

Requires Myron L bluDock™ accessory package, Model # BLUDOCK. Package includes Ultrameter II hardware modification that allows the unit to communicate wirelessly with a personal computer configured for wireless device communication. Package also includes U2CI software application that will operate on Windows XP, Vista and 7*, and Macintosh OSX** based computer systems and Bluetooth USB adapter (dongle) for computers that do not have Bluetooth capability.

A. Software Installation

1. Place Myron L Ultrameter II U2CI Installation CD v2.0.0 & later into your computer or download U2CI application from the Myron L website: http://myronl.com/main/U2CI_Application_DL.htm
2. Upon opening, select the folder for your operating system.
3. Install U2CI application. See detailed installation instructions on CD or Myron L website: http://myronl.com/main/U2CI_Application_DL.htm
4. Additional drivers may be required. See our website for the latest information.

B. Hardware Setup

For a computer without *Bluetooth®* capability:
If you don't have the dongle that came with the BLUDOCK, one can be ordered separately from the Myron L Company. Order Model # BDDO. Plug in your dongle and install per manufacturer's instructions.

For computers with Bluetooth capability/Bluetooth dongle installed:

First time use of the bluDock:

1. Press any parameter button to turn the Ultrameter II on.
2. Put the Ultrameter II in "**PC On**" mode by pressing the (MR ▼) key until "**PC OFF**" appears (see Figure 30).
3. Then press the (CAL MCLR) key. "**PC On**" will be displayed (see Figure 31).

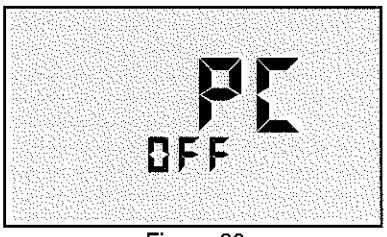


Figure 30

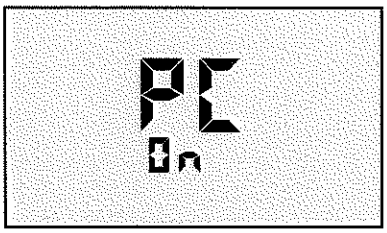


Figure 31

NOTE: "**PC Ini**" may momentarily be displayed while initializing (see Figure 32).

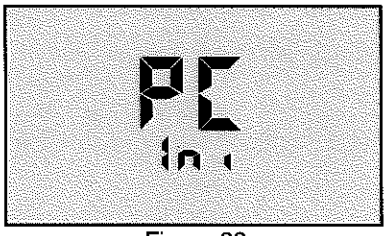


Figure 32

4. Add bluDock to your Bluetooth devices per your operating system procedure. **THE BLUDOCK DEVICE PASSKEY IS 1234.**
5. After pairing, note the number of the COM port assigned by the computer.
   In Windows XP, note the number of the outgoing COM port assigned by the computer.

NOTE: The unit will automatically power down after 60 sec. If the unit powers down during pairing, repeat steps 1-3 above and continue.

C. Memory Stack Download

1. With the Ultrameter II in "**PC On**" mode, open the U2CI software application.
2. Verify that the port selected matches the COM port number noted (first time only). This is the outgoing COM port on Windows XP.
3. In the U2CI application, click on the data download button. A data transfer bar will appear while the data is being downloaded.

Once downloaded, the data may be manipulated, printed or stored within the Myron L U2CI application, or the data may be exported to another more powerful spreadsheet[†] such as Excel*.

[†] Please Note: Although the Myron L Company has performed extensive testing, we cannot guarantee compatibility of all applications and formats. We suggest testing your application and format for compatibility before relying on it.

Additional features such as assigning a name to the unit, setting time and date and erasing data are available. See U2CI software installation CD or visit our website for the latest instructions:
http://myronl.com/main/U2CI_Application_DL.htm

4. Upon completion, click on the "disconnect" icon.
5. Turn off Ultrameter II PC download mode by selecting any measurement function. Failure to do so will reduce battery life.

* Windows 2000, 2007, XP & Vista are registered trademarks of Microsoft Corporation.

** Macintosh OS9.2 & OSX are registered trademarks of Apple Computer Inc.

XVII. CARE and MAINTENANCE

Ultrameter IIs should be rinsed with clean water after use. Solvents should be avoided. Shock damage from a fall may cause instrument failure.

A. Temperature Extremes

Solutions in excess of 71°C/160°F should not be placed in the cell cup area; this may cause damage. The pH sensor may fracture if the Ultrameter II temperature is allowed to go below 0°C/32°F. Care should be exercised not to exceed rated operating temperature.

Leaving the Ultrameter II in a vehicle or storage shed on a hot day can easily subject the instrument to over 66°C/150°F. This will void the warranty.

B. Battery Replacement

Dry Instrument THOROUGHLY. Remove the four (4) bottom screws. Open instrument carefully. Carefully detach battery from circuit board. Replace with 9 volt alkaline battery. Replace bottom, ensuring the sealing gasket is installed in the groove of the top half of case. Re-install screws, tighten evenly and securely.

NOTE: *Because of nonvolatile EEPROM circuitry, all data stored in memory and all calibration settings are protected even during power loss or battery replacement. However, loss of time and date may occur if battery is removed for longer than 3 minutes (180 seconds).*

C. pH/ORP Sensor Replacement (6Psi)

Order model RPR. When ordering, be sure to include the model and serial number of your instrument to ensure receipt of the proper type. Complete installation instructions are provided with each replacement sensor.

D. Cleaning Sensors

1. Conductivity/TDS/Resistivity

The conductivity cell cup should be kept as clean as possible. Flushing with clean water following use will prevent buildup on electrodes. However, if very dirty samples — particularly scaling types — are allowed to dry in the cell cup, a film will form. This film reduces accuracy. When there are visible films of oil, dirt, or scale in the cell cup or on the electrodes, use isopropyl alcohol or a foaming non-abrasive household cleaner. Rinse out the cleaner and your Ultrameter II is again ready for accurate measurements.

2. pH/ORP (6Psi)

The unique pH/ORP sensor in your Ultrameter II is a nonrefillable combination type that features a porous liquid junction. *It should not be*

allowed to dry out. However, if this occurs, the sensor may sometimes be rejuvenated by first cleaning the sensor well with Isopropyl alcohol or a liquid spray cleaner such as Windex™ or Fantastic™ and rinsing well. Do not scrub or wipe the pH/ORP sensor.

Then use one of the following methods:

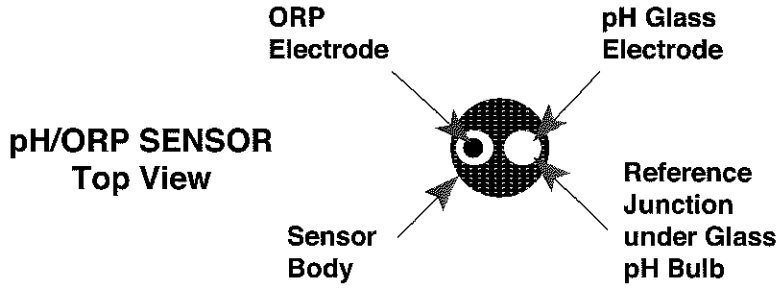
1. Pour a HOT salt solution ~60°C/140°F — a potassium chloride (KCl) solution such as Myron L pH/ORP Sensor Storage Solution is preferable, but HOT tap water with table salt (NaCl) will work fine — in the sensor well and allow to cool. Retest.

   or

2. Pour DI water in the sensor well and allow to stand for no more than 4 hours (longer can deplete the reference solution and damage the glass bulb). Retest.

If neither method is successful, the sensor must be replaced.

"Drifting" can be caused by a film on the pH sensor bulb and/or reference. Use isopropyl alcohol (IPA) or spray a liquid cleaner such as Windex™ or Fantastic™ into the sensor well to clean it. The sensor bulb is very thin and delicate. Do not scrub or wipe the pH/ORP sensor.



Leaving high pH (alkaline) solutions in contact with the pH sensor for long periods of time is harmful and will cause damage. Rinsing such liquids from the pH/ORP sensor well and refilling it with Myron L Storage Solution, a saturated KCl solution, pH 4 buffer, or a saturated solution of table salt and tap water, will extend the useful life.

Samples containing chlorine, sulfur, or ammonia can "poison" any pH electrode. If it is necessary to measure the pH of any such sample, thoroughly rinse the sensor well with clean water immediately after taking the measurement. Any sample element that reduces (adds an electron to) silver, such as cyanide, will attack the reference electrode.

Replacement sensors are available only from the Myron L Company or its authorized distributors (see Replacement pH/ORP Sensor (6Psi), pg. 37).

XVIII. TROUBLESHOOTING CHART

Symptom	Possible Cause
No **display**, even though measurement key pressed	Battery weak or not connected.
Inaccurate **pH** readings (6Psi)	1. pH calibration needed. Ref. pH Cal., pg. 17. 2. Cross-contamination from residual pH buffers or samples in sensor well. 3. Calibration with expired pH buffers.
No response to **pH** changes (6Psi)	Sensor bulb is cracked or an electromechanical short caused by an internal crack.
Will not adjust down to **pH** 7 (6Psi)	pH/ORP sensor has lost KCl.
pH readings drift or respond slowly to changes in buffers/samples *or* "**FAC**" is displayed repeatedly (6Psi)	1. Temporary condition due to "memory" of solution in pH sensor well for long periods. 2. Bulb dirty or dried out. 3. Reference junction clogged or coated.
Unstable **Conductivity/TDS/ Resistivity** readings	1. Dirty electrodes. 2. Test samples greater than 1 megohm.
Unable to calibrate **Conductivity/TDS**	Film or deposits on electrodes.
Resistivity readings much lower than expected	1. Contamination from previous sample or from pH sensor well. 2. Carbon dioxide in test sample.

Corrective Action
Check connections or replace battery. Ref. Battery Replacement, pg. 32.
1. Recalibrate instrument. 2. Thoroughly rinse sensor well. 3. Recalibrate using fresh buffers. Ref. pH Buffer Solutions, pg. 36.
Replace pH/ORP sensor. Ref. Replacement pH/ORP Sensor, pg. 37.
Clean and rejuvenate sensor (ref. Cleaning Sensors, pg. 32) and recalibrate. If no improvement, replace pH/ORP sensor (ref. Replacement pH/ORP Sensor, pg. 37).
Clean and rejuvenate sensor (ref. Cleaning Sensors, pg. 32) and recalibrate. If no improvement, replace pH/ORP sensor (ref. Replacement pH/ORP Sensor, pg. 37).
1. Clean cell cup and electrodes. Ref. Cleaning Sensors, pg. 32. 2. Minimize test sample exposure to air. Ref. Measuring Resistivity, pg. 10.
Clean cell cup and electrodes. Ref. Cleaning Sensors, pg. 32.
1. Rinse cell cup more thoroughly before measurement. Ensure pH cap is snugly in place. 2. See Measuring Resistivity, pg. 10.

XIX. ACCESSORIES

A. Conductivity/TDS Standard Solutions

Your Ultrameter II has been factory calibrated with the appropriate Myron L Company NIST traceable KCl, NaCl, and our own 442™ standard solutions. Most Myron L conductivity standard solution bottles show three values referenced at 25°C: Conductivity in microsiemens/ micromhos, the ppm/TDS equivalents (based on our 442 Natural Water™) and NaCl standards. All standards are within ±1.0% of reference solutions. *Available in 2 oz., quarts/liters, and gallon/~3.8 liter bottles.*

1. Potassium Chloride (KCl)

The concentrations of these reference solutions are calculated from data in the International Critical Tables, Vol. 6. The 7000 μS is the recommended standard. *Order KCL-7000*

2. 442 Natural Water™

442 Natural Water Standard Solutions are based on the following salt proportions: 40% sodium sulfate, 40% sodium bicarbonate, and 20% sodium chloride, which represent the three predominant components (anions) in freshwater. This salt ratio has conductivity characteristics approximating fresh natural waters and was developed by the Myron L Company over four decades ago. It is used around the world for measuring both conductivity and TDS in drinking water, ground water, lakes, streams, etc. 3000 ppm is the recommended standard.
Order 442-3000

3. Sodium Chloride (NaCl)

This is especially useful in sea water mix applications, as sodium chloride is the major salt component. Most Myron L standard solution labels show the ppm NaCl equivalent to the conductivity and to ppm 442 values. The 14.0 mS is the recommended standard. *Order NACL-14.0*

B. pH Buffer Solutions (6Psi)

pH buffers are available in pH values of 4, 7 and 10. Myron L Company buffer solutions are traceable to NIST certified pH references and are color-coded for instant identification. They are also mold inhibited and accurate to within ±0.01 pH units @ 25°C. Order 4, 7 or 10 Buffer.
Available in 2 oz., quarts/liters, and gallon/~3.8 liter bottles.

C. pH Sensor Storage Solution (6Psi)

Myron L pH Sensor Storage Solution prolongs the life of the pH sensor.
Available in 2 oz., quarts/liters, and gallon/~3.8 liter bottles.

D. Soft Protective Carry Cases

Padded Nylon carrying case features a belt clip for hands-free mobility.

Two colors to choose from;
Blue - Model #: UCC
Desert Tan - Model #: UCCDT

E. Hard Protective Carry Cases

Large case with 2 oz. bottles of calibration standard solutions (KCl-7000, 442-3000, 4, 7, & 10 pH buffers and pH storage solution).
Model #: PKUU
Small case (no calibration standard solutions) - *Model #: UPP*

F. Replacement pH/ORP Sensor (6Psi)

pH/ORP sensor is gel filled and features a unique porous liquid junction. It is user-replaceable and comes with easy to follow instructions.
Model #: RPR

G. bluDock™ Wireless Data Transfer Accessory Package

This accessory allows the operator to download the Ultrameter II memory stack to a spreadsheet on a computer. The package includes bluDock modified circuit board in the unit, software CD, installation and operating instructions, and dongle.
Model #: BLUDOCK

XX. TEMPERATURE COMPENSATION (Tempco) of Aqueous Solutions

Electrical conductivity indicates solution concentration and ionization of the dissolved material. Since temperature greatly affects ionization, conductivity measurements are temperature dependent and are normally corrected to read what they would be at 25°C.

A. Standardized to 25°C

Conductivity is measured with great accuracy in the Ultrameter II using a method that ignores fill level, electrolysis, electrode characteristics, etc., and features a microprocessor to perform temperature compensation. In simpler instruments, conductivity values are usually assigned an average correction similar to that of KCl solutions for correction to 25°C. The correction to an equivalent KCl solution is a standard set by chemists that standardizes the measurements and allows calibration with precise KCl solutions. In the Ultrameter II, this correction can be set to other solutions or tailored for special measurements or applications.

B. Tempco Variation

Most conductivity instruments use an approximation of the temperature characteristics of solutions, perhaps even assuming a constant value. The value for KCl is often quoted simply as 2%/°C. In fact, KCl tempco

varies with concentration and temperature in a non-linear fashion. Other solutions have more variation still. The Ultrameter II uses corrections that change with concentration and temperature instead of single average values. See Chart 1.

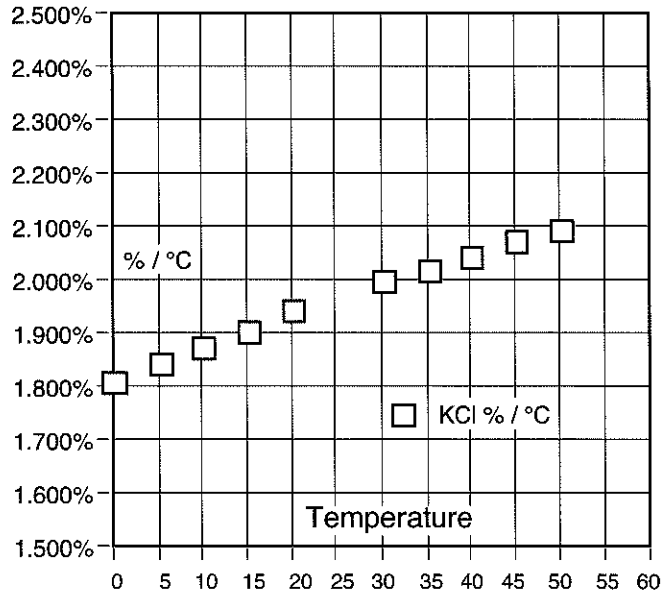


Chart 1

C. <u>An Example of 2 different solution selections and the resulting compensation</u>

How much error results from treating natural water as if it were KCl at 15°C?

A tap water solution should be compensated as 442 with a tempco of 1.68 %/°C, where the KCl value used would be 1.90 %/°C.

Suppose a measurement at 15°C/59°F is 900 microsiemens of true uncompensated conductivity.

Using a 442 correction of 10 (degrees below 25) x 1.68% indicates the solution is reading 16.8% low. For correction, dividing by (.832) yields 1082 microsiemens as a compensated reading.

A KCl correction of 10 (degrees below 25) x 1.9% indicates the solution is reading 19% low. Dividing by (.81) yields 1111 microsiemens for a compensated reading. The difference is 29 out of 1082 = 2.7%.

D. A Chart of Comparative Error

In the range of 1000 μS, the error using KCl on a solution that should be compensated as NaCl or as 442, is illustrated in the graph below.

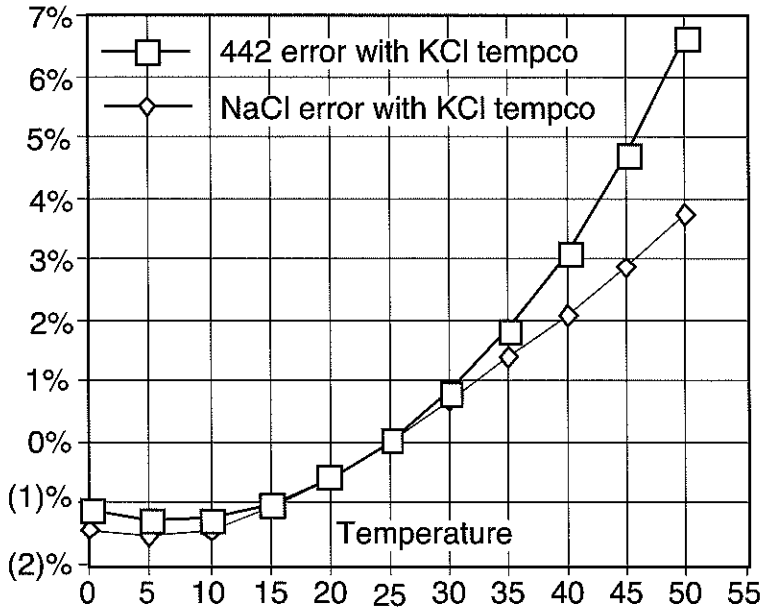


Chart 2

Users wanting to measure natural water based solutions to 1% would have to alter the internal compensation to the more suitable preloaded "442" values or stay close to 25°C. Users who have standardized to KCl-based compensation may want to stick with it, regardless of increasing error as you get further from 25°C. The Ultrameter II will provide the repeatability and convertibility of data necessary for relative values for process control.

E. Other Solutions

A salt solution like sea water or liquid fertilizer acts like NaCl. An internal correction for NaCl can be selected for greatest accuracy with such solutions. Many solutions are not at all similar to KCl, NaCl or 442. A sugar solution, or a silicate, or a calcium salt at a high or low temperature may require a "User" value peculiar to the application to provide readings close to the true compensated conductivity.

Clearly, the solution characteristics should be chosen to truly represent the actual water under test for rated accuracy of ±1%. Many industrial applications have historically used relative measurements seeking a

number to indicate a certain setpoint or minimum concentration or trend. The Ultrameter II gives the user the capability to collect data in "KCl conductivity units" to compare to older published data, in terms of NaCl or 442, or as appropriate. The Ultrameter II can be used to reconcile data taken with other compensation assumptions, especially with its ability to allow custom characteristics through the "User" mode.

XXI. CONDUCTIVITY CONVERSION to TOTAL DISSOLVED SOLIDS (TDS)

Electrical conductivity indicates solution concentration and ionization of the dissolved material. Since temperature greatly affects ionization, conductivity measurements are temperature dependent and are normally corrected to read what they would be at 25°C (ref. Temperature Compensation, pg. 37).

A. How it's Done

Once the effect of temperature is removed, the compensated conductivity is a function of the concentration (TDS). Temperature compensation of the conductivity of a solution is performed automatically by the internal processor with data derived from chemical tables. Any dissolved salt at a known temperature has a known ratio of conductivity to concentration. Tables of conversion ratios referenced to 25°C have been published by chemists for decades.

B. Solution Characteristics

Real world applications have to measure a wide range of materials and mixtures of electrolyte solutions. To address this problem, industrial users commonly use the characteristics of a standard material as a model for their solution, such as KCl, which is favored by chemists for its stability.

Users dealing with sea water, etc., use NaCl as the model for their concentration calculations. Users dealing with freshwater work with mixtures including sulfates, carbonates and chlorides, the three predominant components (anions) in freshwater that the Myron L Company calls "natural water". These are modeled in a mixture called "442™" which the Myron L Company markets for use as a calibration standard, as it does standard KCl and NaCl solutions.

The Ultrameter II contains algorithms for these 3 most commonly referenced compounds. The solution type in use is displayed on the left. Besides KCl, NaCl, and 442, there is the "User" choice. The benefit of "User" is that one may enter the temperature compensation and TDS ratio by hand, greatly increasing accuracy of

readings for a specific solution. That value remains a constant for all measurements and should be reset for different dilutions or temperatures.

C. When does it make a lot of difference?

First, the accuracy of temperature compensation to 25°C determines the accuracy of any TDS conversion. Assume we have industrial process water to be pretreated by RO. Assume it is 45°C and reads 1500 μS uncompensated.

1. If NaCl compensation is used, an instrument would report 1035 μS compensated, which corresponds to 510 ppm NaCl.

2. If 442 compensation is used, an instrument would report 1024 μS compensated, which corresponds to 713 ppm 442.

   The difference in values is 40%.

In spite of such large error, some users will continue to take data in the "NaCl" mode because their previous data gathering and process monitoring was done with an older NaCl referenced device.

Selecting the correct Solution Type on the Ultrameter II will allow the user to attain true TDS readings that correspond to evaporated weight. If none of the 3 standard solutions apply, the "User" mode must be used. Temperature Compensation (Tempco) and TDS Derivation below, details the "User" mode.

XXII. TEMPERATURE COMPENSATION (Tempco) and TDS DERIVATION

The Ultrameter II contains internal algorithms for characteristics of the 3 most commonly referenced compounds. The solution type in use is displayed on the left. Besides KCl, NaCl, and 442, there is the "User" choice. The benefit of "User" mode is that one may enter the tempco and TDS conversion values of a unique solution via the keypad.

A. Conductivity Characteristics

When taking conductivity measurements, the Solution Selection determines the characteristic assumed as the instrument reports what a measured conductivity would be if it were at 25°C. The characteristic is represented by the tempco, expressed in %/°C. If a solution of 100 μS at 25°C increases to 122 μS at 35°C, then a 22% increase has occurred

over this change of 10°C. The solution is then said to have a tempco of 2.2 %/°C.

Tempco always varies among solutions because it is dependent on their individual ionization activity, temperature and concentration. This is why the Ultrameter II features mathematically generated models for known salt characteristics that also vary with concentration and temperature.

B. Finding the Tempco of an Unknown Solution

One may need to measure compensated conductivity of some solution unlike any of the 3 standard salts. In order to enter a custom fixed tempco for a limited measurement range, enter a specific value through the "User" function. The tempco can be determined by 2 different methods:

1. Heat or cool a sample of the solution to 25°C, and measure its conductivity. Heat or cool the solution to a typical temperature where it is normally measured. After selecting "User" function, set the tempco to 0 %/°C as in Disabling Temperature Compensation, pg. 13 (No compensation). Measure the new conductivity and the new temperature. Divide the % decrease or increase by the 25°C value. Divide that difference by the temperature difference.

2. Heat or cool a sample of the solution to 25°C, and measure its conductivity. Change the temperature to a typical measuring temperature. Set the tempco to an expected value as in User Programmable Temperature Compensation, pg. 12. See if the compensated value is the same as the 25°C value. If not, raise or lower the tempco and measure again until the 25°C value is read.

C. Finding the TDS Ratio of an Unknown Solution

Once the effect of temperature is removed, the compensated conductivity is a function of the concentration (TDS). There is a ratio of TDS to compensated conductivity for any solution, which varies with concentration. The ratio is set during calibration in "User" mode as in User Programmable Conductivity to TDS Ratio, pg. 13. A truly unknown solution has to have its TDS determined by evaporation and weighing. Then the solution whose TDS is now known can be measured for conductivity and the ratio calculated. Next time the same solution is to be measured, the ratio is known.

XXIII. pH and ORP (6Psi)

A. pH (6Psi)

1. pH as an Indicator (6Psi)

pH is the measurement of Acidity or Alkalinity of an aqueous solution. It is also stated as the Hydrogen Ion activity of a solution. pH measures the effective, not the total, acidity of a solution.

A 4% solution of acetic acid (pH 4, vinegar) can be quite palatable, but a 4% solution of sulfuric acid (pH 0) is a violent poison. pH provides the needed quantitative information by expressing the degree of activity of an acid or base.

In a solution of one known component, pH will indicate concentration indirectly. However, very dilute solutions may be very slow reading, just because the very few ions take time to accumulate.

2. pH Units (6Psi)

The acidity or alkalinity of a solution is a measurement of the relative availabilities of hydrogen (H^+) and hydroxide (OH^-) ions. An increase in (H^+) ions increases acidity, while an increase in (OH^-) ions increases alkalinity. The total concentration of ions is fixed as a characteristic of water, and balance would be 10^{-7} mol/liter (H^+) and (OH^-) ions in a neutral solution (where pH sensors give 0 voltage).

pH is defined as the negative logarithm of hydrogen ion concentration. Where (H^+) concentration falls below 10^{-7}, solutions are less acidic than neutral, and therefore are alkaline. A concentration of 10^{-9} mol/liter of (H^+) would have 100 times less (H^+) ions than (OH^-) ions and be called an alkaline solution of pH 9.

3. The pH Sensor (6Psi)

The active part of the pH sensor is a thin glass surface that is selectively receptive to hydrogen ions. Available hydrogen ions in a solution will accumulate on this surface and a charge will build up across the glass interface. The voltage can be measured with a very high impedance voltmeter circuit; the dilemma is to connect the voltmeter to solution on each side.

The glass surface encloses a captured solution of potassium chloride holding an electrode of silver wire coated with silver chloride. This is the most inert connection possible from a metal to an electrolyte. It can still produce an offset voltage, but using the same materials to connect to the solution on the other side of the membrane causes the 2 equal offsets to cancel.

The problem is, on the other side of the membrane is an unknown test solution, not potassium chloride. The outside electrode, also called the Reference Junction, is of the same construction with a porous plug in place of a glass barrier to allow the junction fluid to contact the test solution without significant migration of liquids through the plug material. Figure 33 shows a typical 2 component pair. Migration does occur, and this limits the lifetime of a pH junction from depletion of solution inside the reference junction or from contamination. The junction may be damaged if dried out because insoluble crystals may form in a layer, obstructing contact with test solutions. See pH and ORP, pg. 43.

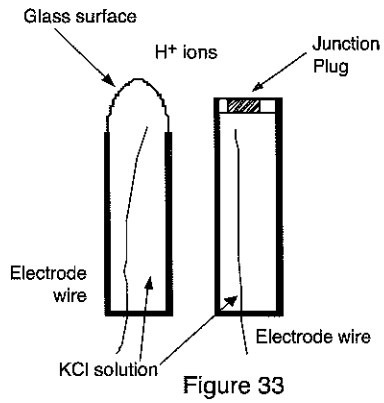


Figure 33

4. The Myron L Integral pH Sensor (6Psi)

The sensor in the Ultrameter II (see Figure 34) is a single construction in an easily replaceable package. The sensor body holds an oversize solution supply for long life. The reference junction "wick" is porous to provide a very stable, low permeable interface, and is located under the glass pH sensing electrode. This construction combines all the best features of any pH sensor known.

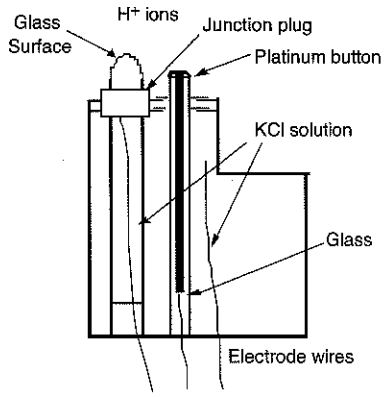


Figure 34

5. Sources of Error (6Psi)

The basics are presented in pH and ORP, pg. 43.

a. Reference Junction

The most common sensor problem will be a clogged junction because a sensor was allowed to dry out. The symptom is a drift in the "zero" setting at 7 pH. This is why the Ultrameter II 6Psi does not allow more than 1 pH unit of offset during calibration. At that point the junction is unreliable.

b. Sensitivity Problems

Sensitivity is the receptiveness of the glass surface. A film on the surface can diminish sensitivity and cause a long response time.

c. Temperature Compensation

pH sensor glass changes its sensitivity slightly with temperature, so the further from pH 7 one is, the more effect will be seen. A pH of 11 at 40°C would be off by 0.2 units. The Ultrameter II 6Psi senses the sensor well temperature and compensates the reading.

B. ORP/Oxidation-Reduction Potential/REDOX (6Psi)

1. ORP as an Indicator (6Psi)

ORP is the measurement of the ratio of oxidizing activity to reducing activity in a solution. It is the potential of a solution to give up electrons (oxidize other things) or gain electrons (reduce).

Like acidity and alkalinity, the increase of one is at the expense of the other, so a single voltage is called the Oxidation-Reduction Potential, with a positive voltage showing, a solution wants to steal electrons (oxidizing agent). For instance, chlorinated water will show a positive ORP value.

2. ORP Units (6Psi)

ORP is measured in millivolts, with no correction for solution temperature. Like pH, it is not a measurement of concentration directly, but of activity level. In a solution of only one active component, ORP indicates concentration. Also, as with pH, a very dilute solution will take time to accumulate a readable charge.

3. The ORP Sensor (6Psi)

An ORP sensor uses a small platinum surface to accumulate charge without reacting chemically. That charge is measured relative to the solution, so the solution "ground" voltage comes from a reference junction - same as the pH sensor uses.

4. The Myron L ORP Sensor (6Psi)

Figure 34, pg. 44, shows the platinum button in a glass sleeve. The same reference is used for both the pH and the ORP sensors. Both pH and ORP will indicate 0 for a neutral solution. Calibration at zero compensates for error in the reference junction.

A zero calibration solution for ORP is not practical, so the Ultrameter II 6Psi uses the offset value determined during calibration to 7 in pH calibration (pH 7 = 0 mV). Sensitivity of the ORP surface is fixed, so there is no gain adjustment either.

5. Sources of Error (6Psi)

The basics are presented in pH and ORP, pg. 43, because sources of error are much the same as for pH. The junction side is the same, and though the platinum surface will not break like the glass pH surface, its protective

glass sleeve can be broken. A surface film will slow the response time and diminish sensitivity. It can be cleaned off with detergent or acid, as with the pH glass.

XXIV. SOFTWARE VERSION

Contact the Myron L Company to see if a software upgrade is available.

1. Press (COND) key.
2. Press (MR ▼) key until three numbers are displayed as shown in Figure 35.
3. Press (COND) key, instrument will time out in ~15 seconds.

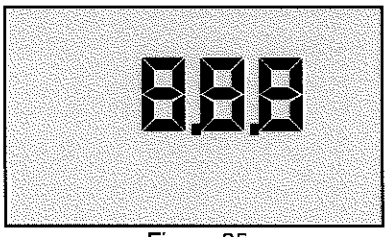


Figure 35

XXV. GLOSSARY

Anions	Negatively charged ions. See Solution Characteristics, pg. 40.
Algorithm	A procedure for solving a mathematical problem. See Temperature Compensation (Tempco) and TDS Derivation, pg. 41.
Logarithm	An arithmetic function. See pH Units, pg. 43.
ORP	Oxidation-Reduction Potential or REDOX, See ORP/ Oxidation-Reduction Potential/REDOX, pg. 45.
TDS	Total Dissolved Solids or the Total Conductive Ions in a solution. See Conductivity Conversion to Total Dissolved Solids (TDS), pg. 40.
Tempco	Temperature Compensation See Temperature Compensation of Aqueous Solutions, pg. 37.
User	A mode of operation that allows the instrument user (operator) to set a tempco and/or a TDS factor for their specific solution type. See Temperature Compensation of Aqueous Solutions, pg. 37 and Temperature Compensation (Tempco) and TDS Derivation, pg. 41.

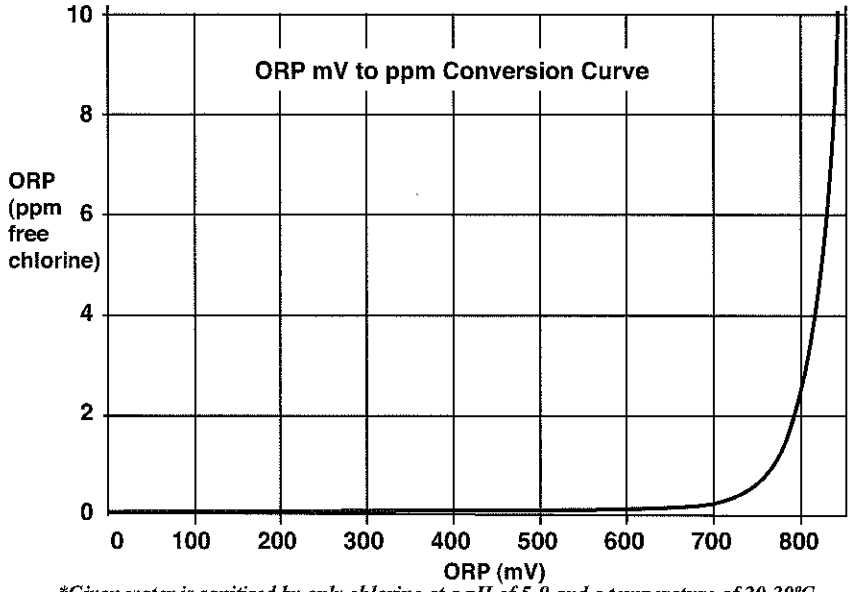
For details on specific areas of interest refer to the Table of Contents.

XXVI. ADDENDUM: NEW ORP PPM FREE CHLORINE FEATURE For 6Psi ONLY Software V.3.51 and later (see pg. 46). Contact the Myron L Company for more information: www.myronl.com

A convenient new feature has been added to the Ultrameter II 6Psi™ that allows the user to choose between ORP millivolts (mV) and parts per million (ppm) free chlorine readings. mV and ppm free chlorine are the two most commonly used sanitizer measurement units used in water quality management. The 6Psi mV to ppm free chlorine conversion algorithm is based on a published conversion curve that assumes a pH of 5-9 and a temperature of 20-30°C in water sanitized by chlorine only and bench testing performed at the Myron L Company. We have increased the accuracy of low-range ppm free chlorine readings by extrapolating from the published data. With this new feature the 6Psi can measure a dynamic range of sanitizer concentrations that is wider than the range of a colorimetric test kit.

Additional ORP Specifications	
Spec	**Value**
Ranges	**± 999 mV** **0.20 – 9.99 ppm**
Resolution	**mV resolution: 1 mV** **ppm resolution: 0.01 ppm**
Accuracy	**± 1 mV** **± 2.5% of reading ppm***

Plot Showing the Conversion Curve between ppm and mV*



**Given water is sanitized by only chlorine at a pH of 5-9 and a temperature of 20-30°C*

NOTE: FOR COMPLETE INSTRUCTIONS ON MEASURING ORP, REFER TO MEASURING ORP, PG. 10, IN THE OPERATION MANUAL.

To change the ORP measurement format preference:

1. Press (ORP).

2. Press and hold (CAL/MCLR) for approximately three seconds.

   The current preference for ORP units of measure is displayed. Factory setting for this preference is mV. (Shown below in Figure 36.)

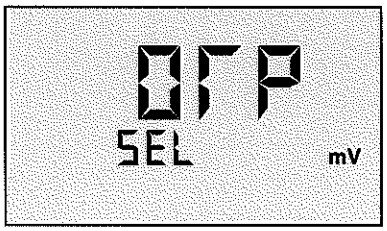


Figure 36

4. Press the (▲/MS) or (MR/▼) keys to toggle between mV and ppm free chlorine measurement format. The setting chosen is displayed (ppm shown as example in Figure 37).

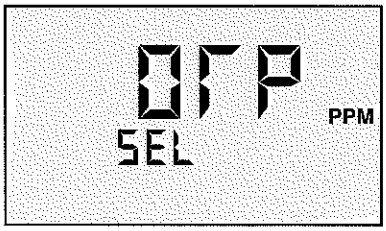


Figure 37

5. Press any parameter key to exit ORP unit preference selection or let the unit time out. ORP unit preference will be saved.

XXVII. ADDENDUM: NEW LSI & HARDNESS CALCULATOR (6Psi only)

The new LSI calculator makes it easy to analyze the scaling nature of water in the lab or in the field. Using a calcium carbonate saturation index algorithm developed by Dr. Wilfred Langelier in 1936, the calculator computes the saturation index of a sample based on measured and inferred values for pH, temperature, hardness and alkalinity. You can then change any of these values in "User" adjust mode to analyze the effect of the change on water balance. "User" mode also allows you to input measured values for alkalinity and hardness as determined by other independent testing for a precise saturation index value.

The 6Psi LSI calculator computes a saturation index value using the following formula:

$$SI = PH + TF + CF + AF - 12.1$$

Where:
PH is the measured value of pH in pH units as determined by the 6Psi
$TF = 0.0117 \times Temp - 0.4116$
$CF = 0.4341 \times \ln(Hrd) - 0.3926$
$AF = 0.4341 \times \ln(AL) - 0.0074$

The following is a general industry guideline for interpreting LSI values:

- An index between -0.5 and +0.5 indicates balanced water
- An index of more than +0.5 indicates scale-forming water
- An index below -0.5 indicates corrosive water

How to Use the 6Psi LSI Calculator

In LSI Calculator mode, accept all 6Psi variable values to obtain the 6Psi saturation index value. Once the 6Psi saturation index value is displayed, you can enter "User" mode to change values for pH, temperature, hardness (ppm or grains), and alkalinity (ppm $CaCO_3$), then recalculate the saturation index based on those changes. The most accurate way to generate a saturation index value is to accept 6Psi measured values for pH and temperature and input values in "User" mode for hardness and alkalinity based on titration testing.

NOTE: You can exit the LSI calculator at any time by pressing any of the parameter keys or by letting the unit time out.

A. LSI Calculator Mode

1. Press any parameter key to turn the instrument on.

2. To access the LSI calculator, press (MR ▼) repeatedly or hold down (fast scroll) until "**CALC LSI**" is displayed (see Figure 38).

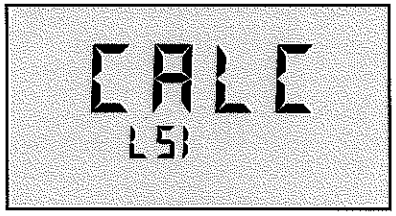


Figure 38

3. Press (CAL/MCLR). "**FILL LSI**" will display (see Figure 39).

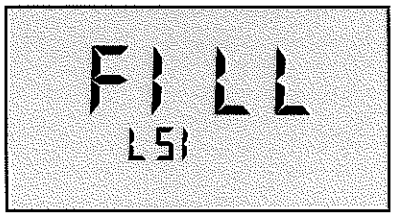


Figure 39

4. Rinse both the pH/ORP sensor well and the conductivity cell cup three times with sample solution.

5. Fill both the sensor well and cell cup with sample solution and press (CAL/MCLR).

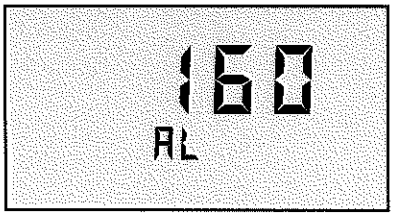


Figure 40

6. The 6Psi inferred value for alkalinity and "**AL**" will display (see example in Figure 40).

   Press (CAL/MCLR).

7. The 6Psi inferred value for hardness and "**Hrd**" will display (see example in Figure 41).

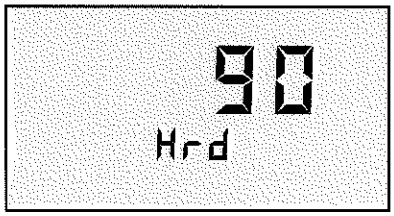


Figure 41

   Press (CAL/MCLR).

8. The pH value measured by the 6Psi and "**PH**" will display (see example in Figure 42).

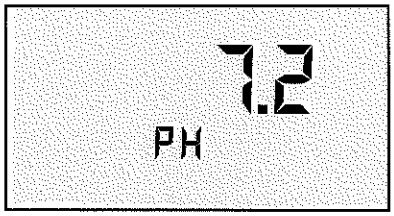


Figure 42

   Press (CAL/MCLR).

9. The temperature value measured by the 6Psi and "**°C**" or "**°F**" will display (see example in Figure 43).

Press (CAL/MCLR).

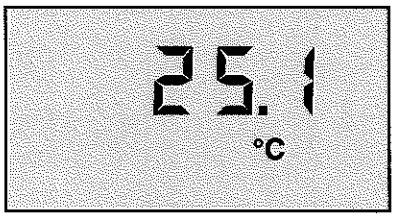


Figure 43

10. The 6Psi generated LSI value will display (see example in Figure 44).

To store this LSI value in memory, press (▲/MS).

If you do not wish to change any of the variable values, exit the LSI calculator by pressing any parameter key or by letting the unit time out.

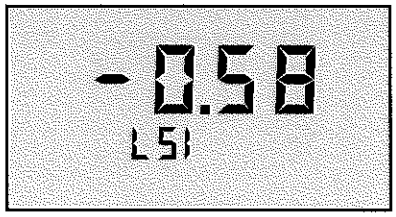


Figure 44

B. LSI Calculator "User" Mode

LSI Calculator "User" mode allows you to change any or all of the variable values then recalculate the saturation index using the adjusted value(s).
NOTE: You can only enter "User" mode AFTER 6Psi LSI value is displayed.

1. Press (CAL/MCLR) after the 6Psi generated LSI value is displayed.

2. "**LSI AdJ**" will display (see Figure 45).

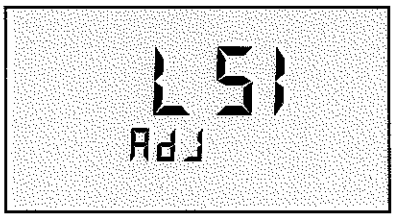


Figure 45

Press (CAL/MCLR).

The 6Psi inferred value for alkalinity and "**AL**" will display (see example in Figure 46).

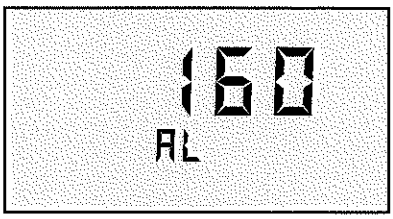


Figure 46

3. Press (▲/MS) or (MR/▼) to adjust the displayed value to the desired value.

Press (CAL/MCLR) to accept the value displayed.

The 6Psi inferred value for hardness and "**Hrd**" will display (see example in Figure 47).

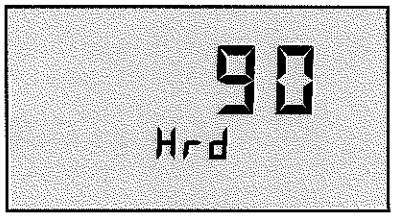


Figure 47

4. Press the (▲/MS) or (MR/▼) keys to adjust the displayed value to the desired value.

Press (CAL/MCLR) to accept the value displayed.

The pH value measured by the 6Psi and "**PH**" will display (see example in Figure 48).

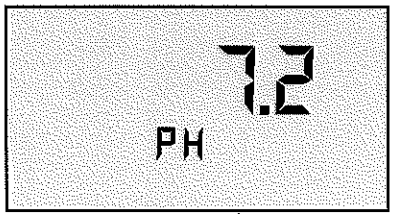


Figure 48

5. Press the (▲/MS) or (MR/▼) keys to adjust the displayed value to the desired value.

Press (CAL/MCLR) to accept the value displayed.

The value for temperature measured by the 6Psi and "**°C**" or "**°F**" will display (see example in Figure 49).

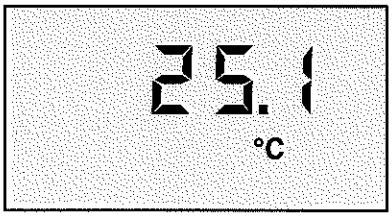


Figure 49

6. Press the (▲/MS) or (MR/▼) keys to adjust the displayed value to the desired value.

Press (CAL/MCLR) to accept the value displayed.

"**User**", the user adjusted LSI value and "**LSI**" will display (see example in Figure 50).

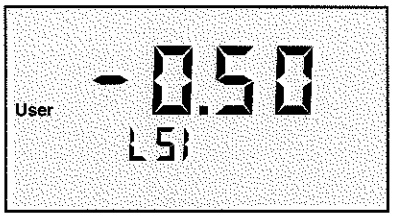


Figure 50

C. Hardness Unit Preference Selection

The LSI Calculator allows you to select either ppm or grains of hardness units for saturation index calculation.

The hardness unit conversion is based on the following equivalency:

17.1 ppm (mg/L) = 1 grain

The hardness range is limited to 0.0 - 1710 ppm and 0.0 - 100 grains of hardness in the 6Psi.

NOTE: The 6Psi MUST be in the hardness screen (**Hrd**) in LSI Calculator mode or LSI Calculator "User" mode for hardness unit preference selection.

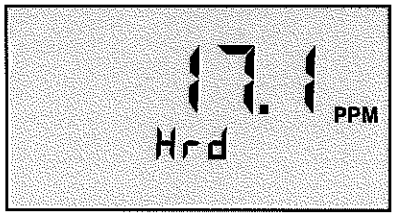


Figure 51

By default, hardness unit preference is set to ppm (see Figure 51).

1. While in the **Hrd** screen in CALC LSI or LSI Adj mode, press and hold the CAL/MCLR key. **HArd SEL** will display along with the current unit preference (see Figure 52).

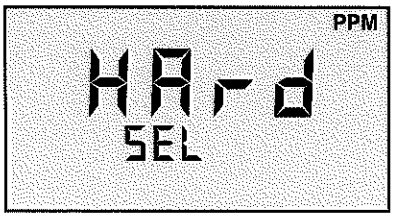


Figure 52

2. Press ▲/MS or MR/▼ to toggle between ppm and grains of hardness. No unit icon is lit when grains is selected (see Figure 53).

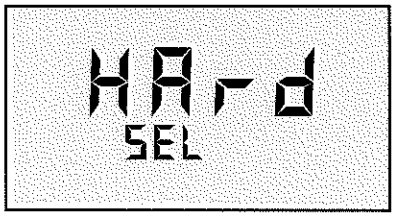


Figure 53

3. Press CAL/MCLR to accept the change. The LSI calculator will resume in the **Hrd** screen. The unit change will be reflected on the display (see Figure 54).

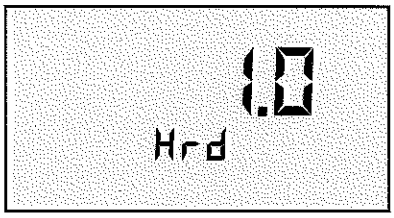


Figure 54

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www.myronl.com

Made In USA

UMIIOM 08SE10

Attachment C – Groundwater Purging and Sampling Log Form

WSP Services Inc.
Groundwater Purging and Sampling Log

Well ID _____ Date _____ Job / task #: _____

Sample ID _____ Site Name: _____

Field Conditions _____

Well Diameter (inches) _____

Measured Well Depth (feet) _____

W.L. Before Purge _____

Date/Time _____

W.L. After purge _____

Date/Time _____

W.L. Time of Sampling _____

Date/Time _____

W.L. for 80% recovery _____

well depth - [(well depth - depth to water) x 0.8] = 80%

Low-Flow Purge Calculations

One system volume = _____(ml)

(One system volume equals the volume of the flow through cell* plus the volume of the discharge tubing** plus the volume of the bladder***)

*flow through cell volume _____ (ml) + *** bladder volume _____(ml)

discharge tubing length _____ (ft)

discharge tubing diameter _____(in.)

**discharge tubing volume = length (ft)_____ x CF = _____(ml)

conversion factors (CF)

1 foot of 3/4 inch diameter tubing equals 86.85 ml

1 foot of 1/2 inch diameter tubing equals 38.51 ml

1 foot of 1/4 inch diameter tubing equals 9.64 ml

Purge Rate _____

Purge Device _____

Purge Method _____

Purge Start Time _____

Pump Depth At _____

Calculated Purge Volume = (_____ - _____) x M = _____ gal.

(well depth - depth to water) x multiplier = Purge Vol.

Actual Volume Purged_____ gal

1 gallon = 3.7853 liters

Purge Volume Multipliers (M)

Casing Dia.	1 Casing Vol.	3 Casing Vol.
2.0	0.16	0.49
4.0	0.65	1.96
4.5	0.83	2.48
6.0	1.47	4.41

QA/QC Information

	X if Present	Designation
Trip blank		
Duplicate		
Field blank		
MS/MSD		
Other		

Time	Purged Volume Cumulative (gal)	Temp. (°C)	Elec. Cond. (µS/cm)	pH	D.O. (mg/l)	ORP (mV)	Turbidity (NTU)	Purge Rate (ml/min)	Depth to Water (feet)

Sample Date/Time: _____ Sample Device: _____

Laboratory Name and Location: _____ Subcontractor: _____

Sampled by: _____ _____

Name and Company *Signature*